COMPARISON OF COMPRESSIVE STRENGTH TEST PROCEDURES FOR BLENDED CEMENTS

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I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.

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ABSTRACT

COMPARISON OF COMPRESSIVE STRENGTH TEST PROCEDURES FOR BLENDED CEMENT

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The aim of this thesis is to twofold, in order to demonstrate the variabilities that can be faced within the compressive strength of blended cements, one blended cement namely CEM IV / B (P-V) 32.5N is selected and the 28-day compressive strength is obtained by 16 different laboratories following TS EN 196-1 standard. Later, to show the variabilities that could be faced by different standards, three different cement types were selected and their compressive strengths are determined following two procedures first with TS EN 196-1, later with similar procedure described in ASTM.

The strength of cement is determined by TS EN 196-1 in Turkey that is the same for all types of cements. However, American cement producers use different standards for testing the strength of Portland cement and blended cements. The main difference is the amount of water utilized in producing the cement mortar.

It was observed that for Portland and Portland composite cements; there is not any significant difference in between the compressive strength results of cement mortars prepared by both methods. However, for pozzolanic cements, there is much deviance in the compressive strength results of cement mortars prepared by TS EN 196-1.

Keywords: Compressive Strength, Inter-laboratory test comparison, Reproducibility and Repeatability, Mortar

ÖZ

KATKILI ÇİMENTO İÇİN BASINÇ DAYANIM DENEY YÖNTEMLERİNİN KARŞILAŞTIRILMASI

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Bu tezin amacı, katkılı çimentoların basınç dayanımında karşılaşılan değişkenliği göstermek için CEM IV / B (P-V) 32.5N tip bir katkılı çimento seçilerek 28 günlük basınç dayanımı TS EN 196-1 standardına uygun olarak 16 laboratuvarda gerçekleştirilmesiyle ikiye ayrılmaktadır. Sonra, farklı standardlarla karşılaşılabilecek değişiklikleri göstermek amacıyla 3 farklı çimento seçilmiş ve bunların basınç dayanımları 2 yönteme göre, ilki TS EN 196-1, ikincisi ATSM'de belirtilen yönteme benzer şekilde belirlenmiştir.

Türkiye'de çimentonun dayanımı tüm çimento tipleri için aynı olan TS EN 196-1 ile belirlenmektedir. Ancak, Amerikan çimento üreticileri portland ve katkılı çimentoların dayanım tayini için farklı Standardlar kullanmaktadırlar. Temel farklılık çimento harcı oluşturulurken kullanılan su miktarındadır.

Bu çalışma sonucunda, Portland ve Portland kompoze çimentolarda iki yöntemi kullanılarak elde edilen harçların basınç dayanım sonuçları arasında hiçbir farklılık olmadığı görülmüştür. Ancak, Puzolanik çimentolarda TS EN 196-1 yöntemiyle elde edilen harçların basınç dayanım sonuçlarında çok fazla sapmalar olduğu gözlenmiştir.

Anahtar Sözcükler: Basınç Dayanımı, Laboratuvarlar Arası Karşılaştırma, Tekrar Edilebilirlik ve Tekrar Üretilebilirlik, Harç

To my father,

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ABBREVIATIONS

AoC : Attestation of Conformity

ASTM : American Society for Testing Materials

COV : Coefficient of Variance

CPD : Construction Products Directive

EN : European Norm

FPC : Factory Production Control

ITT : Initial Type Test

METU : Middle East Technical University

PSD : Particle Size Distribution

TCMA : Turkish Cement Manufacturers’ Association

TCMA – CQE : Turkish Cemet Manufacturers’ Association – Council for Quality and Environment Economic Enterprise

TOC : Total Organic Carbon

TS : Turkish Standard

XRF : X-Ray fluorescence

CHAPTER 1

INTRODUCTION

1.1. General

The cement industry is one of the important components of Turkish economy. According to yearly statistics obtained from Turkish Cement Manufacturers' Association (TCMA) as shown in Figure 1.1, cement production reached 54 Mt in 2009, it increases day by day as the demand for civil infrastructures and buildings such as houses, offices increases.

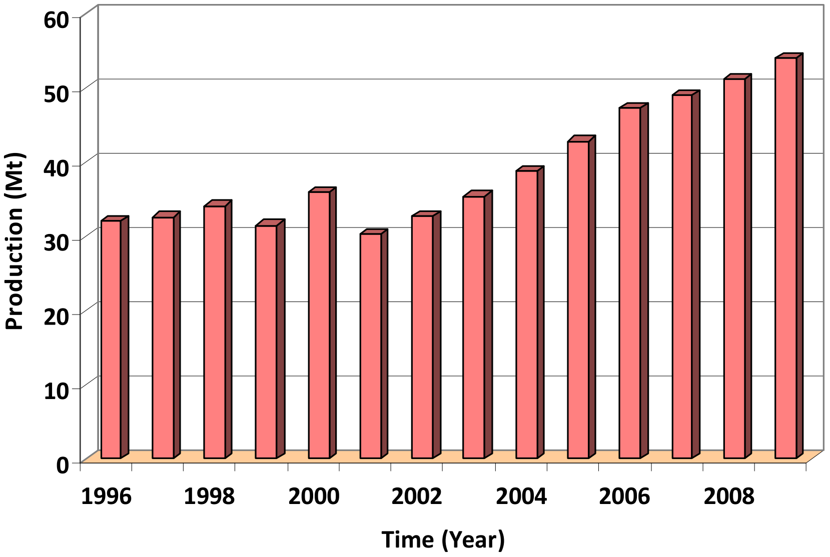


Figure 1.1 Yearly Cement Production in Turkey [1]

Cement sector has been one of the most energy intensive industries in the world. In order to produce one tone of cement, 60 to 130 kg of fuel oil or its equivalent and about 105 kWh of electricity are required depending on the cement type and its production process [2]. Thus, the trend is to use environmental friendly products. Since the cement sector consumes natural resources, the key objective for cement manufacturers is to achieve the environmental sustainability. To achieve this goal, a range of regulations and policies, foster resource efficient and eco-friendly products and raise consumer awareness in Europe is put in force [3]. Due to the regulations, policies and environmental sustainability, cement producers have made several changes on their processes and product ranges. Nowadays, cement manufacturers are utilizing additives, mostly pozzolanic in nature, to decrease the clinker content of the cement. In general, cements produced with additives are called blended cements.

Utilizing additives in cement production may have influences on the cement properties and thus, its quality. In the past, Turkey was frequently subjected to earthquakes and thousands of people were killed or injured during the earthquakes. Poor quality construction materials are one of the reasons for these incidents. Since the cement is an important constituent of concrete, quality of cement also plays an important role in preventing these looses. Therefore, there is a necessity to check and enhance the properties of the blended cements.

The strength of cement is determined by TS EN 196-1 in Turkey that is the same for all types of cements. However, American cement producers use different standards for testing the strength of Portland cement and blended cements. The main difference is the amount of water utilized in producing the cement mortar.

1.2. Objective and Scope of the Thesis

This study consists of two parts. The main objective is to show the discrepancies in cement compressive strength testing standards used for various types of cements. In the first part, blended cement called CEM IV / B (P-V) 32.5N was tested in 16 laboratories according to TS EN 196-1. Water to cement ratio was set to 0.5. The aim of this part is to determine whether the test method used to determine the compressive strength of blended cement is appropriate.

In the second part, two different compressive strength test methods were applied to three different types of cements; namely CEM I 42.5R, CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R. The first method is the same as described in TS EN 196-1. Whereas in the second method the ASTM standards, in which the slump flow was set at a constant value, for blended cements was followed. Therefore, mortars having different water to cement ratio were tested. The objective of this part is to compare the test methods and to determine the best test method for blended cements.

Within the scope of this study, this thesis consists of six chapters:

In Chapter 2, the history of cement is explained briefly. Then, since the quality of cement is important, the quality control application for cement is outlined. In addition, main constituents used in the cement production and their effects on the properties of cement are briefly mentioned. Moreover, the factors affecting compressive strength of concrete and finally, the importance of flow on cement mortar and concrete are described.

In Chapter 3, the inter-laboratory test evaluation and the statistical analysis program used in this study are described. In addition, an inter laboratory test evaluation organized by Turkish Cement Manufacturers’ Association Council for Quality and Environment Economic Enterprise is given.

In Chapter 4, the properties of materials used in the study and the details of the tests performed on the samples are given. Moreover, the experimental program along with the mixture ratio is provided.

In Chapter 5, compressive strength test results of cement types CEM I 42.5R, CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R and their inter-laboratory test evaluations are presented. Data are discussed. Also, statistical analysis performed on the compressive strength of the cements is given.

Chapter 6 presents the conclusions of the study resulting from the findings of the tests, observations, and the recommendations to future researchers.

CHAPTER 2

THEORETICAL CONSIDERATIONS

2.1. Portland Cement

Cement is a binder which mainly consists of compounds of calcium, silicium, aluminum, iron and small amounts of other materials. The cements used in concrete production are called hydraulic cements which set and harden after being combined with water.

In the earliest of the 19th century, Joseph Aspdin, a bricklayer, first made a hydraulic cement called Portland cement whose name was given since the hardened cement resembles the color and quality of Portland stone [4]. Since then, Portland cement is produced by intimately mixing together calcareous and argillaceous, or other silica-, alumina-, and iron oxide bearing materials, burning them in a kiln at a temperature of about 1450°C, and grinding the resulting clinker with a small amount (3-5%) of gypsum [5].

There are many types of cements defined in different standards. In the harmonized Turkish standard TS EN 197-1, there are 27 different main types of cement and 6 different strength classes for cement which totally makes 162 possible cement types. This standard covers both for Portland and blended cements. Whereas in American standards three types of standards exist; one is for various types of Portland cement ASTM C 150, second is for blended cements ASTM C 595 and third is for a broad performance based specs ASTM C 1157.

2.2 Quality Control Applications in Cement

Cement is one of the construction products mentioned in Council Directive 89/106/EEC, i.e. Construction Products Directive (CPD). In order to provide free movement of cement throughout the whole European countries, a mark called CE Mark, the notation of French word phrase “Conformité Européenné” demonstrating the product satisfies the requirements set by harmonized national laws and regulations, must be affixed also in Turkey where this European Directive is also put into force in the scope of EU harmonization process [6]. However, several conformity applications are performed whether it is suitable to affix CE Mark to these products or not.

In general, for the attestation of conformity (AoC) of construction products, there are four different conformity control systems namely 1, 1+, 2, 2+, 3 and 4 as presented in Table 2.1. According to these AoC systems, responsibilities are defined for manufacturers or both for manufacturers and notified bodies. The responsibilities are defined regarding to the risks of the products. For example, products within the AoC system 1+ have the highest risk, whereas the products within the AoC system 4 have the lowest risk [7].

Cement, being a transportable construction product, is evaluated in the AoC system 1+. Thus, there is an independent and impartial body notified by European Commission, which is called notified body, takes place in the attestation of conformity [7]. In this type of AoC system, by notified bodies, not only the factory and factory production control are checked, but also quality of the final product is checked by taking samples from the factory.

Table 2.1 Attestation of Conformity Systems Requirements [7]

		Attestation of Conformity Systems					
		1+	1	2+	2	3	4
Tasks for Manufacturers	Factory Production Control (FPC)	✓	✓	✓	✓	✓	✓
	Initial Type Test (ITT) of Product			✓	✓		✓
	Testing of Spot Samples According to Test Plan	✓	✓	✓			
Tasks for Notified Bodies	Initial Inspection of Factory and FPC	✓	✓	✓	✓		
	ITT of Product	✓	✓			✓	
	Continuous Surveillance of Factory and FPC	✓	✓	✓			
	Taking Audit Samples from the Factory	✓					

The initial type test sample as the name implies is the first sample according to which free movement of the cement is determined. Until the results conforming TS EN 197-1 of this sample is obtained, it is forbidden to affix CE Mark to the product and also sell it in the European market [7]. After permission to affix CE Mark to the product, there is a 3 month period defined for cement in TS EN 197-2 as initial period for a new type of cement. During the initial period, the frequencies of the samples taken by both the manufacturer and the notified body are higher when compared with the routine period.

Initial period of cement ends up according to results of the conformity evaluation of the initial period. If the results of the evaluation confirm the requirements of TS EN 197-1, then initial period ends up and as routine period of 12 months starts.

2.2.1 Conformity Evaluation of Cement

As seen in Table 2.1, both the manufacturer and the notified body take samples from the factory. The samples taken by the manufacturer according to its test plan are called as autocontrol samples. The samples taken by the notified body are called as audit samples. Autocontrol samples are tested only by manufacturer, whereas audit samples are tested by both the manufacturer and the notified body [6].

The quality control of these cement samples are performed in accordance with the standards TS EN 197-1 and TS EN 197-2. The former defines the required parameters for cement and describes statistical analysis performed for these parameters. Whereas, the latter defines the factory production control and describes statistical analysis performed to check the reliability of the standard compressive strength, i.e. 28 day compressive strength, results [6].

In TS EN 197-1, the required mechanical and physical properties of cement regardless of its type are given in Table 2.2. The determinant parameters for these requirements are the early and standard strength classes of the cement.

As seen in Table 2.2, there are two different early compressive strength classes; namely, ordinary early compressive strength denoted by “N” and higher early compressive strength denoted by “R”. For standard compressive strength, there are three different compressive strength classes, namely 32.5, 42.5 and 52.5 [6]. For all of the standard compressive strength classes, the early compressive strength equals to the 2 day compressive strength, except for cement having the standard compressive strength as 32.5. For this class of cement, the early compressive strength equals to 7 day compressive strength.

For early compressive strength and initial setting time, there is only lower limit defined for each class in TS EN 197-1. For compressive strength both lower and upper limits exist. However, for soundness, there is only one upper limit defined for all strength classes.

Table 2.2 Mechanical and Physical Requirements of Cement [6]

Strength Class	Compressive Strength MPa				Initial Setting Time	Soundness (Expansion)
	Early Strength		Standard Strength			
	2 day	7 day	28 day		min	mm
32.5 N	-	≥ 16.0	≥ 32.5	≥ 52.5	≥ 75	≤ 10
32.5 R	≥ 10.0	-				
42.5 N	≥ 10.0	-	≥ 42.5	≥ 62.5	≥ 60	
42.5 R	≥ 20.0	-				
52.5 N	≥ 20.0	-	≥ 52.5	-	≥ 45	
52.5 R	≥ 30.0	-				

Moreover, the chemical requirements of cement, except for sulfate content, are defined according to its type as seen in Table 2.3. For sulfate content both cement type and strength class determine the required values.

Table 2.3 Chemical Requirements of Cement [6]

Property	Test Reference	Cement Type	Strength Class	Requirements a)
Loss on Ignition	EN 196-2	CEM I CEM III	all	≤ 5.0 %
Insoluble Residue	EN 196-2 b)	CEM I CEM III	all	≤ 5.0 %
Sulfate Content (as SO_3)	EN 196-2	CEM I CEM II c) CEM IV CEM V	32.5 N 35.2 R 42.5 N	≤ 3.5 %
			42.5 R 52.5 N 52.5 R	≤ 4.0 %
		CEM III d)	all	
Chloride Content	EN 196-21	all e)	all	≤ 0.10 % f)
Pozzolanicity	EN 196-5	CEM IV	all	Satisfies the Test

a) Requirements are given as percentage by mass of the final cement.
b) Determination of residue insoluble in hydrochloric acid and sodium carbonate.
c) Cement type CEM II/B-T may contain up to 4.5 % sulfate for all strength classes.
d) Cement type CEM III/C may contain up to 4.5 % sulfate.
e) Cement type CEM III may contain more than 0.10 % chloride but in that case the maximum chloride content shall be stated on the packaging and/or the delivery note.
f) For pre-stressing applications cements may be produced according to a lower requirement. If so, the value of 0.10 % shall be replaced by this lower value which shall be stated in the delivery note.

Manufacturers should take samples in accordance with their test plan. The minimum testing frequency of this test plan is also defined in TS EN 197-1 and given in Table 2.4. The required tests that shall be applied to the sample according to its type, the corresponding test method and the statistical analysis procedure are given in Table 2.4 both for cement in routine and initial period. The minimum testing frequency for initial period is approximately two times the minimum testing frequency for routine period. Moreover, the highest number of samples are obtained for compressive strength, initial setting and sulfate content both for initial and routine period [6]. In initial period, the highest number of samples is obtained also for expansion test.

Table 2.4 Minimum Testing Frequency for the Autocontrol Tests and Statistical Analysis Procedure [6]

			Autocontrol Testing			
			Minimum Testing Frequency		**Statistical Assessment Procedure**	
					Inspection by	
Property	**Cements to be tested**	**Test method** [a] [b]	**Routine Situation**	**Initial Period for a new type of cement**	**Variables** [e]	**Attributtes**
Early Strength Standard Strength	All	EN 196-1	2/week	4/week	x	-
Initial Setting Time	All	EN 196-3	2/week	4/week	-	x [f]
Soundness (Expansion)	All	EN 196-3	1/week	4/week	-	x
Loss on Ignition	CEM I, CEM III	EN 196-2	2/month [c]	1/week	-	x [f]
Insoluble Residue	CEM I, CEM III	EN 196-2	2/month [c]	1/week	-	x [f]
Sulfate Content	All	EN 196-2	2/week	4/week	-	x [f]
Chloride Content	All	EN 196-21	2/month [c]	1/week	-	x [f]
Pozzolanicity	CEM IV	EN 196-5	2/month	1/week	-	
Composition	All	- [d]	1/month	1/week	-	x

[a] Where allowed in the relevant part of EN 196, other methods than those indicated may be used provided they give results correlated and equivalent to those obtained with the reference method.

[b] The methods used to take and prepare samples shall in accordance 50 % EN 196-7.

[c] When none of the test results within a period of 12 months exceeds 50 % of the characteristics value the frequency may be reduced to one per month.

[d] Appropriate test method chosen by the manufacturer.

[e] If the data are not normally distributed then the method of assessment may be decided on a case by case basis.

[f] If the number of samples is at least one per week during the control period, the assessment may be made by variables.

For cement, besides the difference in between testing frequency of manufacturer in initial and routine period, there is also a difference in between testing frequency of notified body in the initial and routine periods. In TS EN 197-2, the minimum testing frequency for audit samples are defined as one sample per every month for 3 months during the initial period. Thus, for the conformity evaluation of cement in the initial period, there must be at least 3 audit samples and 52 autocontrol samples. However, the minimum testing frequency for audit samples in a routine period is defined as at least six samples per year [7]. Thus, for the conformity evaluation of cement in routine period, a total of at least 6 audit samples and 104 autocontrol samples are required [6].

In the statistical analysis of cement according to TS EN 197-1 and TS EN 197-2, the more emphasis is given to the compressive strength since the compressive strength is one of the most important properties of cements. Most of the nonconformities are resulting from the compressive strength test results.

2.2.1.1. Statistical Analysis According to TS EN 197-1

Statistical conformity analysis is performed according to two different methods, namely inspection by variables and inspection by attributes, as defined in TS EN 197-1 both for initial and routine period. As mentioned earlier, the period of the conformity analysis is 3 months for initial period and 12 months for routine period. As stated in TS EN 197-2, for routine period, conformity analysis is performed 2 times in a year.

In general, evaluation of compressive strength results is performed in accordance with the “inspection by variables”. Although there is a chance for the manufacturers to choose the inspection method of cement properties other than compressive strength, they prefer the inspection method of these properties to be “inspection by attributes”.

2.2.1.1.2 Inspection by Variables

It is assumed that the test results are normally distributed. According to inspection by variables, conformity is achieved when the test results satisfy the following equations [6]:

$$\bar{x} - k_A \times SD \geq \text{Lower Limit} \qquad \textbf{(2.1)}$$

and;

$$\bar{x} + k_A \times SD \leq \text{Upper Limit} \qquad \textbf{(2.2)}$$

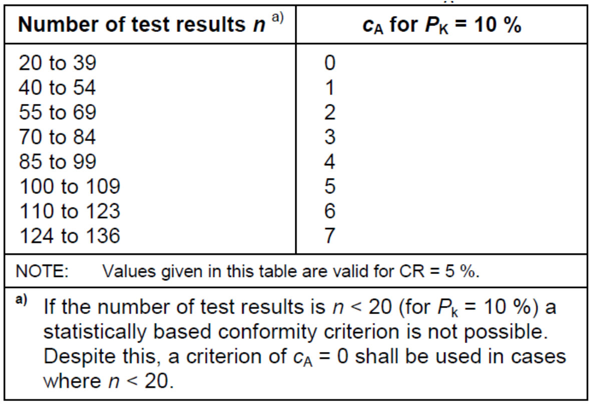
where $\bar{x}$ is the arithmetic mean of the autocontrol test results in the control period; SD is the standard deviation of the autocontrol test results in the control period; k_A is a statistical constant depends on the number of samples. Lower and upper limits are specified in Table 2.2. Since there is only a lower limit for early compressive strength and standard compressive strength class of 52.5, the conformity evaluations of these two classes with respect to inspection by variables are performed only by Equation 2.2.

2.2.1.1.2 Inspection by Attributes

Inspection by attributes is performed for the properties of cement other than compressive strength.

In inspection by attributes, test results are compared with the characteristic values given in Tables 2.2 and 2.3. The values outside its corresponding characteristic values, denoted by c_D, are counted. According to number of autocontrol samples, allowable number of errors c_A is determined from Table 2.5. P_k in the table represent the percentile on which the specified characteristic value is based.

Table 2.5 Allowable Number of Test Results Outside The Characteristic Value, c_A [6]



Number of test results n [a)]	c_A for P_K = 10 %
20 to 39	0
40 to 54	1
55 to 69	2
70 to 84	3
85 to 99	4
100 to 109	5
110 to 123	6
124 to 136	7
NOTE: Values given in this table are valid for CR = 5 %.	
[a)] If the number of test results is $n < 20$ (for P_k = 10 %) a statistically based conformity criterion is not possible. Despite this, a criterion of $c_A = 0$ shall be used in cases where $n < 20$.	

Conformity is demonstrated, when the test results satisfy the Equation 2.3.

$$c_D \leq c_A \tag{2.3}$$

where c_D is the number of test results outside the characteristic value given in Tables 2.2 and 2.3, c_A is the allowable number of test results outside the characteristic value which depends on the number of samples.

2.2.1.2 Statistical Analysis According to TS EN 197-2 Annex A

Statistical analysis of autocontrol test results is defined in TS EN 197-1. Meanwhile, statistical analysis of three data sets composed of autocontrol samples, audit samples of manufacturer and notified body results is defined in TS EN 197-2 Annex A. In this analysis, only 28 day compressive strength results are examined and representativeness and the accuracy of these data sets are evaluated.

Three data sets included in this analysis are [6, 8];

- A Series- Autocontrol test results of the manufacturer
- B Series- Audit samples test results of the manufacturer
- C Series- Audit samples test results of the notified body

The analysis consists of:

- Comparison of A and B series (Control of sampling error)
- Comparison of B and C series (Control of experimental error)

Note that these two comparison analyses can only be performed provided that there are at least six audit samples. Therefore, since there are three audit samples in the initial period, performing this analysis is not appropriate.

In A and B series comparison, series are checked whether they belong to the same population or not. In order to say that A and B series belong to the same population; Equation 2.4 must be satisfied [8]:

$$\left|MA - MB\right| \leq 2.0\,MPa \quad \textbf{(2.4)}$$

where MA is the arithmetic mean of the autocontrol test results, and MB is the arithmetic mean of the manufacturers audit test results. If the difference between arithmetic mean of A and B satisfies Equation 2.4, then these two data series belong to the same population.

Although, in some situations, absolute value of the difference between arithmetic mean of A and B might not satisfy Equation 2.4, it is not appropriate to conclude that these data series do not belong to the same population. Therefore, for such situations, there is another equation to calculate and compare A and B series given in TS EN 197-2 Annex A as;

$$\left|MA - MB\right| \leq 2.58 \times S_A / \sqrt{N_B} \quad \textbf{(2.5)}$$

where S_A is the standard deviation of the autocontrol test results, N_B is the number of audit samples.

If the difference between arithmetic mean of A and B do not satisfy Equation 2.5, within a 99% confidence level, A and B data series belong to different populations.

There are several reasons that may lead to non-conformity in A and B comparison. They are given as follows:

- If samplers do not apply the test method as described, for example mixing times and/or amount of ingredients are not the same as in the test method,
- If different samplers performing the tests of autocontrol and audit samples, there might be personnel errors leading the unconformity,
- If the test method applied is not appropriate, consequently the materials are not properly mixed,
- If there is a fluctuation in the production so that the target margins of the product always change,
- If the autocontrol and audit samples do not belong to the same type of cement, then deviations in between A and B series will occur.

In B and C series comparison, series are examined to control the accuracy of the autocontrol sample results. In order to ensure the accuracy, both of the following two equations must be satisfied [8].

$$\mathrm{SD} \leq 3.4 \qquad \textbf{(2.6)}$$

$$\left|\mathrm{MB} - \mathrm{MC}\right| \leq 4.0\,\mathrm{MPa} \qquad \textbf{(2.7)}$$

where SD is the standard deviation of the difference between the corresponding results of the audit samples as defined by $d_i=B_i-C_i$, MB is the arithmetic mean of the manufacturer’s audit sample, MC is the arithmetic mean of the notified body’s audit samples. If either or both of the Equations 2.6 and 2.7 are not satisfied, then according to TS EN 197-2 Annex A, both the manufacturer and notified body must investigate the reasons.

There are several reasons that may lead to non-conformity in B and C comparison. They are given as follows:

- Although the test method applied by the samplers of manufacturer and notified body is the same, since the samplers are not the same, there might be some personnel errors,
- If the test method is not properly applied, for example mixing times and/or amount of ingredients are not the same as in the test method,
- If the test method applied is not appropriate, consequently the materials are not properly mixed, then deviations in between B and C series will occur.
- Although the same parameters are set for the testing machine in TS EN 196-1, according to the usage frequency of the testing machine, there might be some deviations resulting from the testing machine.

2.3 Main Constituents of Cement

The main cementitious material in concrete is cement. However, to decrease the cost, to improve the concrete performance and to produce more environmental friendly products several supplementary cementitious materials, which are generally natural minerals or by-products of some other industrial processes, are used in cement to produce what is generally called as blended cements [6]. Thus, cement consists of different materials, which are homogenous in composition. The main constituents of cement other than clinker listed in TS EN 197-1 are as follows:

- ➢ Pozollanic materials
    - Natural Pozzolana (P)
    - Artificial Pozzolana

    - Silica fume (D)
    - Granulated Blast Furnace Slag (S)
    - Fly Ash (V, W)
        - Siliceous fly ash (V)
        - Calcareous fly ash (W)
    - Burnt shale (T)
- Limestone (L, LL)

The percentage of these materials in cements varies according to cement type and the application and the properties of concrete desired. However, TS EN 197-1 states that the composition of the cements confirming this standard shall be in the limits given in Table 2.6.

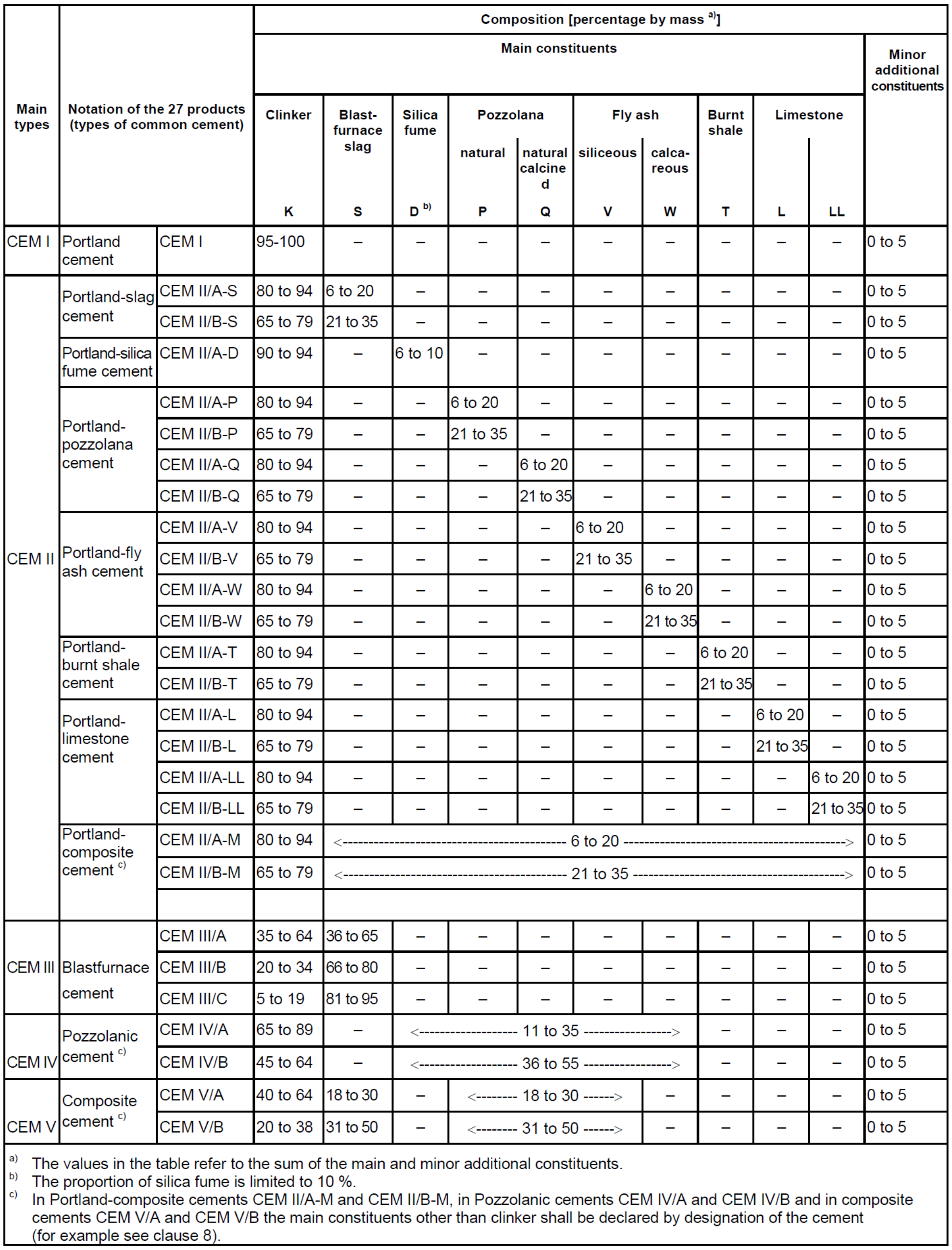
2.3.1 Pozzolanic Materials

Pozzolanic materials do not harden in themselves when mixed with water. They exhibit cementitious properties when combined with calcium hydroxide at ambient temperatures [9].

2.3.1.1 Natural Pozzolan (P, Q)

Materials originated from volcanic eruption are usually called as natural pozzolanas [10]. According to TS EN 197-1, there are two types of natural pozzolana; namely, natural and natural calcined pozzolanas abbreviated by P and Q, respectively.

Table 2.6 Percentage of Cement Composition According to TS EN 197-1 [6].



Main types	Notation of the 27 products (types of common cement)		Composition [percentage by mass [a]]										
			Main constituents										Minor additional constituents
			Clinker	Blast-furnace slag	Silica fume	Pozzolana		Fly ash		Burnt shale	Limestone		
						natural	natural calcined	siliceous	calcareous				
			K	S	D [b]	P	Q	V	W	T	L	LL	
CEM I	Portland cement	CEM I	95-100	–	–	–	–	–	–	–	–	–	0 to 5
CEM II	Portland-slag cement	CEM II/A-S	80 to 94	6 to 20	–	–	–	–	–	–	–	–	0 to 5
		CEM II/B-S	65 to 79	21 to 35	–	–	–	–	–	–	–	–	0 to 5
	Portland-silica fume cement	CEM II/A-D	90 to 94	–	6 to 10	–	–	–	–	–	–	–	0 to 5
	Portland-pozzolana cement	CEM II/A-P	80 to 94	–	–	6 to 20	–	–	–	–	–	–	0 to 5
		CEM II/B-P	65 to 79	–	–	21 to 35	–	–	–	–	–	–	0 to 5
		CEM II/A-Q	80 to 94	–	–	–	6 to 20	–	–	–	–	–	0 to 5
		CEM II/B-Q	65 to 79	–	–	–	21 to 35	–	–	–	–	–	0 to 5
	Portland-fly ash cement	CEM II/A-V	80 to 94	–	–	–	–	6 to 20	–	–	–	–	0 to 5
		CEM II/B-V	65 to 79	–	–	–	–	21 to 35	–	–	–	–	0 to 5
		CEM II/A-W	80 to 94	–	–	–	–	–	6 to 20	–	–	–	0 to 5
		CEM II/B-W	65 to 79	–	–	–	–	–	21 to 35	–	–	–	0 to 5
	Portland-burnt shale cement	CEM II/A-T	80 to 94	–	–	–	–	–	–	6 to 20	–	–	0 to 5
		CEM II/B-T	65 to 79	–	–	–	–	–	–	21 to 35	–	–	0 to 5
	Portland-limestone cement	CEM II/A-L	80 to 94	–	–	–	–	–	–	–	6 to 20	–	0 to 5
		CEM II/B-L	65 to 79	–	–	–	–	–	–	–	21 to 35	–	0 to 5
		CEM II/A-LL	80 to 94	–	–	–	–	–	–	–	–	6 to 20	0 to 5
		CEM II/B-LL	65 to 79	–	–	–	–	–	–	–	–	21 to 35	0 to 5
	Portland-composite cement [c]	CEM II/A-M	80 to 94	<-------------------- 6 to 20 -------------------->									0 to 5
		CEM II/B-M	65 to 79	<-------------------- 21 to 35 -------------------->									0 to 5
CEM III	Blastfurnace cement	CEM III/A	35 to 64	36 to 65	–	–	–	–	–	–	–	–	0 to 5
		CEM III/B	20 to 34	66 to 80	–	–	–	–	–	–	–	–	0 to 5
		CEM III/C	5 to 19	81 to 95	–	–	–	–	–	–	–	–	0 to 5
CEM IV	Pozzolanic cement [c]	CEM IV/A	65 to 89	–	<---------- 11 to 35 ---------->					–	–	–	0 to 5
		CEM IV/B	45 to 64	–	<---------- 36 to 55 ---------->					–	–	–	0 to 5
CEM V	Composite cement [c]	CEM V/A	40 to 64	18 to 30	–	<-------- 18 to 30 ------>			–	–	–	–	0 to 5
		CEM V/B	20 to 38	31 to 50	–	<-------- 31 to 50 ------>			–	–	–	–	0 to 5

[a] The values in the table refer to the sum of the main and minor additional constituents.

[b] The proportion of silica fume is limited to 10 %.

[c] In Portland-composite cements CEM II/A-M and CEM II/B-M, in Pozzolanic cements CEM IV/A and CEM IV/B and in composite cements CEM V/A and CEM V/B the main constituents other than clinker shall be declared by designation of the cement (for example see clause 8).

2.3.1.2 Artificial Pozzolan

Artificial pozzolans are the by-products of various thermal treatments, such as burnt shale, silica fume, fly ash, slag, etc.

2.3.1.2.1 Silica fume (D)

Silica fume, also called condensed silica fume and micro silica, is a finely divided residue resulting from the production of elemental silicium or ferrosilicon alloys that is carried from the furnace by exhaust gases [11].

2.3.1.2.2 Granulated Blast Furnace Slag (S)

In the production of iron, iron ore is smelted in a blast furnace. During this process, molten iron that is collected in the bottom of the furnace and liquid iron blast furnace slag floating on the pool of iron, are periodically tapped from the furnace at a temperature of 1400-1500^{o}C [12]. Granulated blast furnace slag is made by rapid cooling of a slag melt which contains at least two-thirds by mass of glassy slag and has hydraulic properties.

It is stated in TS EN 197-1 that granulated blast furnace slag composition shall have at least two-thirds by mass of the sum of calcium oxide (CaO), magnesium oxide (MgO) and silicon dioxide (SiO_2). The rest of the composition is aluminium oxide (Al_2O_3) together with small amounts of other compounds. Also, (CaO + MgO)/(SiO_2) ratio by mass shall exceed 1.0 [6]

2.3.1.2.3 Fly Ash (V, W)

Fly ash is a finely divided residue that results from the combustion of pulverized coal and is carried from the combustion chamber of the furnace by exhaust gases. Commercially available fly ash is a by-product of thermal power plants [11].

TS EN 197-1 divides fly ashes into two groups; namely, siliceous and calcareous fly ashes.

2.3.1.2.3.1 Siliceous fly ash (V)

Siliceous fly ash, a fine powder of mostly spherical particles having pozzolanic properties, consists mainly of reactive SiO_2 and Al_2O_3 [6].

2.3.1.2.3.2 Calcareous fly ash (W)

Calcareous fly ash, a fine powder having both hydraulic and/or pozzolanic properties, consists mainly of reactive CaO, SiO_2 and Al_2O_3 [6].

2.3.1.2.4 Burnt Shale (T)

Burnt shale is another cementitious constituent used in cement production. Burnt shale is produced by burning of oil shale in fluidized bed furnace at temperatures between 600 and 800^oC and composed of clinker phases, mainly dicalcium silicate and monocalcium aluminate.

2.3.2 Limestone (L, LL)

Limestone, a sedimentary rock, consists mainly of calcium carbonate; the most stable form is calcite. Limestone often contains Mg, Al and Fe combined as carbonates and silicates.

It is stated in TS EN 197-1 that in order to use limestone as a constituent in cement, calcium oxide content should be at least 75% by mass. Moreover, limestone is divided into two groups in TS EN 197-1 according to its Total Organic Carbon (TOC) content. If TOC value does not exceed 0.20 % by mass, the limestone is demonstrated with LL. If TOC value does not exceed 0.50 % by mass, then the limestone is demonstrated with L [6].

2.3.3 Effects of the Mineral Additives on Mortar and Concrete Properties

Mineral additives influence the properties of cements and concretes. The following subsections present the effects of main constituent of cement on water requirement, workability and strength.

2.3.3.1 Water Requirement

The amount of mixing water required for a specified consistency of a mortar or concrete is called as water requirement, determined by mortars, of cement mortar or concrete. Adding excess or less amount of water can lead to adverse results on the strength of cement mortar or concrete. Therefore, it is required to determine how much water is sufficient for the cement mortar or concrete.

Cementitious materials have different impacts on the water requirement of cement mortar or concrete since they have different particle size, shape, particle size distributions etc. For example, natural pozzolans have significant effect on water demand of concrete. Since the natural pozzolans increase the specific surface area, cements containing natural pozzolans have higher water requirement as compared to ordinary portland cement [14]. The same effect is also observed when clinker is replaced with silica fume in cement. Therefore, there is a limit in water requirement in TS EN 197-1, because of the high fineness of silica fume.

However, for a given slump, water requirement of a cement containing fly ash may be less than the water requirement of portland cement. Although the dosage of fly ash increases the water reduction, not all fly ash does the same effect on mortar. Brink and Halstead reported that the water demand increases as the carbon content of the fly ash increases [13].

2.3.3.2 Workability

Workability is defined as the easiness of the concrete mixing, handling, compacting, placing and finishing. Water content of concrete has an important effect on workability. There are several factors affecting workability such as quantity and characteristics of cementing materials, and amount of water etc.

The lubricant effect and morphology improvement on cement mortar or concrete of natural pozzolans increase with an increase in fineness of the cementitious materials [14]. As a result, natural pozzolans improve the consistency and the workability of the concrete. Yijin et al (2004) studied the usage of fly ashes having different fineness as a cementitious material replacing the clinker in cement and replacing cement in concrete [15]. They found out that fly ash improves the workability of cement mortar or concrete due to their spherical shape causing “ball bearing” effect. Also, the water requirement of concrete containing ground granulated blast furnace slag decreases with the increase in the amount of ground granulated blast furnace slag [16].

2.3.3.3 Strength

Supplementary cementitious materials such as fly ash, ground granulated blast furnace slag, burnt shale and silica fume contribute to the strength gain of concrete. However, the characteristics of the supplementary materials and replacement level limit them for the strength gain of concrete [17]. For example, pozzolanic reactivity of the fly ash is one of the limiting parameter.

In addition to cementitious materials used, test type is another factor affecting the strength. As the size of the specimen, moisture content of the specimen, the rate of loading and type of test machine change, the strength results change.

2.4 Factors Affecting Concrete Strength

Concrete is a composite material consisting of mainly cement, water, coarse and fine aggregates and chemical admixtures. Several complex reactions providing the strength gain of concrete take place when these materials are mixed.

There are several factors affecting the strength of concrete such as constituents and their mixing proportions, test method applied for the determination of strength, production method of concrete etc. Some of these factors are given in Figure 2.1.

As the strength of cement is determined on cement mortars prepared by cement, water and sand, the factors affecting concrete strength will also affect the strength of cements.

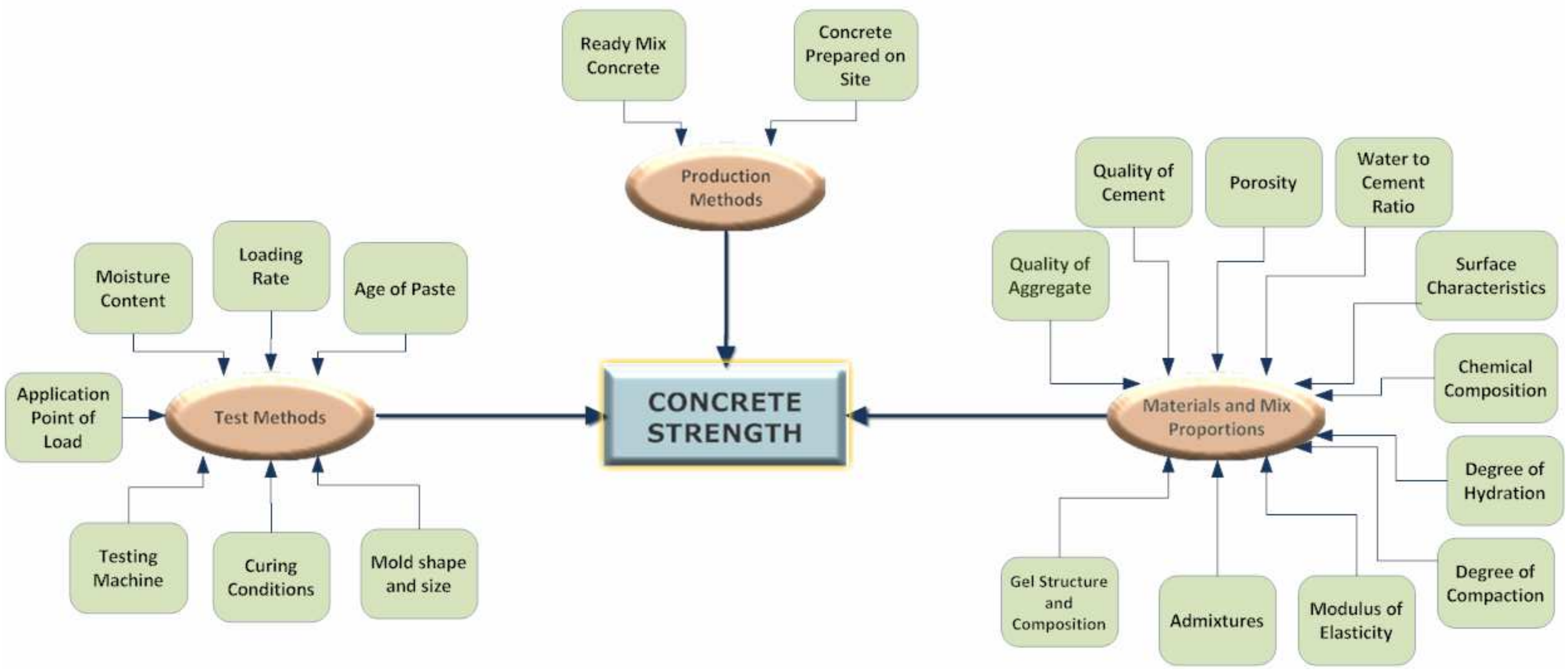


Figure 2.1 Factors Affecting the Strength of Concrete

In the quality control testing of cement, strength is the parameter that causes differences between the audit laboratory and factory laboratory. This may have several reasons such as materials and their mixing proportions and test method.

2.4.1 Materials and Their Mixing Proportions

Since the concrete is a mixture of cement, water, coarse and fine aggregate and chemical admixtures, properties of each of these materials have an influence on the strength of concrete. Cement type, particle size distribution of cement and aggregates are some of the factors related to constituents.

In addition to the properties of the constituents, mixing proportions of these materials influence the strength of concrete. There is an optimum mixing proportions for these materials to produce an economical concrete having the desired properties. For example, amount of water or water to cement ratio are important parameters. Because excessive water results higher bleeding and segregation of concrete and thus, decreases the strength of concrete. Whereas, less amounts of water prevent production of sufficiently homogenous mixes.

2.4.2 Test Method

Different test methods are applied to determine the compressive strength of cement and concrete. For example, size of the specimen, in ASTM C109, 50 mm cubic mold is used for the determination of compressive strength of cement, whereas in TS EN196-1 a 40 × 40 × 160 mm prism is used. Moreover, before the determination of compressive strength of cement, flexural strength is applied to the specimen and thus, the specimen is divided into two. Although, the broken part is not placed directly into the application of the compressive force, there may be impact on the compressive strength of the specimen, because specimen might not be divided into two equally. Therefore, the application point of the force may not be at the center of the specimen which may result in faulty results in the compressive strength.

Yi et al. (2006) observed that for cube and prism specimens the impact of the size is bigger when compared with the cylinder specimens [18]. In addition to that, they have also investigated the correlation between shape and size of the specimen for concretes having normal and high strengths. According to their findings, for high strength concretes, the shape effect of the specimen is approximately negligible when compared to normal strength ones. Moreover, they found out that the strength level impact on the size effect of the specimen increases as the shape effect of the specimen decreases.

Viso et al. (2008) also examined the size effect on the compressive strength of concrete [19]. According to their study, the smaller specimen's resistance to stress is bigger than the larger specimens. Moreover, size of the cubic specimen influences the strength result more when compared with the cylinders.

In addition to size and shape of the specimen, curing conditions of the specimen plays an important role on the compressive strength of concrete. Chemical reactions take place during the hardening of concrete. Therefore, the temperature at which the reactions take place affects the concrete properties.

2.5 Importance of Flow

Flow test, which depends on especially the water to cement ratio and on various aspects of the cement such as fineness, flocculation, and rate of hydration reactions, gives an idea about the consistency of a cement mortar or a fresh concrete [20].

Consistency is an important parameter for the concrete workability. In addition, by using a standard consistency, i.e. using a standard flow, errors because of consolidation or bleeding in samples are avoided [20].

2.6 Inter-Laboratory Test Comparison

An inter-laboratory test is carried out by a representative number of participating laboratories repeatedly within each participating laboratory on identical samples under agreed conditions.

There are three main objectives for inter-laboratory testing:

- Proficiency Testing
- Certification of Materials
- Test Method Validation

The precision, which is a fundamental characteristic of a test method, is the degree to which the repeated tests under the same conditions show the same results. Since an inter-laboratory test is an appropriate procedure to measure the precision of a test method, an inter-laboratory test is applied to test the precision of the test method prescribed in TS EN 196-1.

In addition to determination of confidence interval, calculation of repeatability and the reproducibility of the test results play an important role upon an inter-laboratory test comparison. Guslicov et al. (2009) reported that, the progress of the standard deviation of repeatability and reproducibility and coefficient of variance have given an idea on the progress of the inter-laboratory tests [21]. They noticed that during these 20 years period, as the development of standards and the interpretation and applicability of the standards increase, the coefficient of variance decreases as shown in Figure 2.2. Moreover, since the repeatability and reproducibility values decreased, they concluded that there is an improvement in the applicability of the participants studied on the test method. However, it should be mentioned that these tests were performed on CEM I type of cements.

For compressive strength, which is performed by experienced laboratories under the conditions defined in TS EN 196-1, the reproducibility in terms of coefficient of variance is expected to be less than 6%. The reproducibility in terms of coefficient of variance has reached less than 6% as shown in Figure 2.2.

In addition, in TS EN 196-1, it is stated that the repeatability in terms of coefficient of variance is expected to be in between 1 and 3 %.

Different organizations, i.e. ATILH, CEPROCIM, etc., performed lots of inter-laboratory test comparisons. However, in these inter-laboratory test comparisons, especially for compressive strength comparison, CEM I type cements were usually used. Therefore, there is a need to determine the repeatability of blended cements.

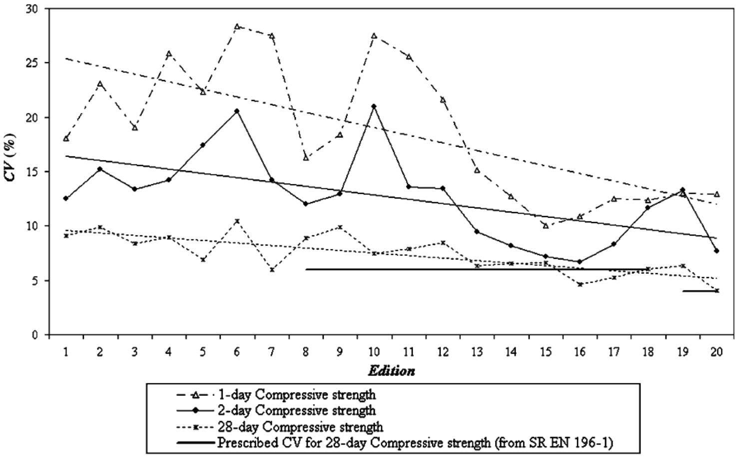


Figure 2.2 Progress of the Coefficient of Variance for 1, 2 and 28-day Compressive Strength [21]

CHAPTER 3

INTER-LABORATORY EVALUATION OF STRENGTH TEST RESULTS

3.1 Inter-Laboratory Test Evaluation Procedure

The assessment of the inter-laboratory test results is carried out in accordance with TS EN 196-1 and Normal Gauss Distribution.

3.1.1 Acceptance of Test Results

Acceptance of test results was determined according to TS EN 196-1. For each mold, if there is any result showing 10% deviance from the mean of the six results, it is discarded. Then, the remaining five results are averaged. If again, there is any result showing 10% deviance from the mean of the five results, all of the results are discarded.

3.1.2 Omission of Outliers

Since the unjustified minimization of the extreme values result in an impression of the performance of the test method, the extreme values, called outliers for each data set, were omitted. In order to determine the outliers of the rest of the data sets, Grubb’s Test was applied. In Grubb’s Test, by ranking the data set, the smallest and the largest values are determined. Then, the mean and the standard deviation are calculated. Depending on the suspicion of a possible outlier of a value, one of the following equations is used [24]:

$$T_{Min} = \frac{x - x_{Smallest}}{\sigma} \tag{3.1}$$

$$T_{Max} = \frac{x_{Largest} - x}{\sigma} \tag{3.2}$$

where x is the mean of the data set, $x_{Smallest}$ is the smallest number in the data set, $x_{Largest}$ is the largest number in the data set, and σ is the standard deviation of the data set.

The calculated T values are compared with the critical values given in Table 3.1. If both the calculated T_{Min} and T_{Max} are less than $T_{Critical}$, then it is concluded that there is no outlier in the data set. However, if one of those values or both of them are greater than $T_{Critical}$, then it is concluded that the result by which a T value greater than $T_{Critical}$, is obtained. That result is marked as an outlier and must be discarded.

Table 3.1 Critical Values for Grubb's Test

n	g_{Crit} α= 0.05	g_{Crit} α= 0.01	n	g_{Crit} α= 0.05	g_{Crit} α= 0.01
3	1.1531	1.1546	15	2.4090	2.7049
4	1.4625	1.4925	16	2.4433	2.7470
5	1.6714	1.7489	17	2.4748	2.7854
6	1.8221	1.9442	18	2.5040	2.8208
7	1.9381	2.0973	19	2.5312	2.8535
8	2.0317	2.2208	20	2.5566	2.8838
9	2.1096	2.3231	21	2.6629	3.0086
10	2.1761	2.4097	22	2.7451	3.1029
11	2.2339	2.4843	23	2.8675	3.2395
12	2.2850	2.5494	24	2.9570	3.3366
13	2.3305	2.6070	25	3.0269	3.4111
14	2.3717	2.6585	26	3.0839	3.4710

However, since the Grubb's test is valid for a data set that are normally distributed, normality of the data set must be checked.

Due to the limited number of samples, a nonparametric test called Kolmogorov-Smirnov Test by using a computer program called Statistical Package for the Social Sciences (SPSS) is applied to determine the normality of the data set.

The hypotheses used in this test are:

> H_O: there is no difference between the distribution of the data set and a normal one
> H_A: there is a difference between the distribution of the data set and normal

The P-value is provided by SPSS. If it is below 0.05, then the data set is determined not to be normally distributed.

3.1.3 Evaluation of the Test Results

After finding out the outliers as described above, the evaluation of the test results were performed. Since sample size was so small the t-test was used to determine the confidence interval. According to the number of data sets included in the assessment, t value corresponding to 95% confidence for two-tail is chosen from t-table given in Table 3.2. Then, 95% confidence interval is calculated with the expression given below.

$$\text{ConfidenceInterval} = \chi \pm t \times \frac{\sigma}{\sqrt{N}} \qquad \textbf{(3.3)}$$

where χ is the arithmetic mean, t is the corresponding *t* value given in Table 3.2, σ is the standard deviation and N is the number of data sets.

Table 3.2 t-Table

The Degree of Freedom	Cumulative Probability For two-tails		The Degree of Freedom	Cumulative Probability For two-tails	
	0.10	0.05		0.10	0.05
1	6.314	12.71	9	1.833	2.262
2	2.920	4.303	10	1.812	2.228
3	2.353	3.182	11	1.796	2.201
4	2.132	2.776	12	1.782	2.179
5	2.015	2.571	13	1.771	2.160
6	1.943	2.447	14	1.761	2.145
7	1.895	2.365	15	1.753	2.131
8	1.860	2.306	16	1.746	2.120

3.1.4 Calculation of the Repeatability and Reproducibility

According to TS 5822-2 ISO 5725-2, accuracy of a test method is determined by its repeatability and reproducibility values [25]. Therefore, the repeatability and reproducibility values are calculated to demonstrate the accuracy of a test method.

As stated in TS 5822-2 ISO 5725-2, when the true value of a standard deviation is not known in statistical practice, it is replaced by an estimate based upon a sample, then the symbol σ is replaced by *s*. Thus, for the obtained results, the standard deviation of repeatability and reproducibility are calculated using Equations 3.4 and 3.7, respectively [25].

$$s_{rj} = \sqrt{\frac{\sum_{i=1}^{p}(n_{ij}-1)s_{ij}^2}{\sum_{i=1}^{p}(n_{ij}-1)}} \qquad \textbf{(3.4)}$$

$$\overset{=}{y}_j = \frac{\sum_{i=1}^{p} n_{ij}\,\bar{y}_{ij}}{\sum_{i=1}^{p} n_{ij}} \qquad \textbf{(3.5)}$$

$$s_{Lj} = \sqrt{\frac{1}{p-1}\sum_{i=1}^{p}(\bar{y}_{ij}-\overset{=}{y}_j)^2 - \frac{s_{rj}^2}{2}} \qquad \textbf{(3.6)}$$

$$s_{Rj} = \sqrt{s_{rj}^2 + s_{Lj}^2} \qquad \textbf{(3.7)}$$

where p denotes the total number of laboratories participating in the inter-laboratory experiment, i denotes the number of a particular laboratory, j denotes the mold number, n denotes the number of test results obtained in one laboratory at one mold, $\bar{y}$ denotes the arithmetic mean of the test results, $\bar{\bar{y}}$ denotes the grand mean of the test results.

Note that s_r is the estimate of the repeatability variance; s_L is the symbol used for the estimate of the between-laboratory variance; s_R is the estimate of the reproducibility variance.

3.2 Comparison of the Test Results by Using Mann-Whitney Test Method

In addition to inter-laboratory test evaluation, test results are compared with each other to determine the similarities. For this purpose, test results are compared among each other by using Mann-Whitney test which is used to compare two groups of sample data.

In Mann-Whitney Test, two data sets are compared whether the data samples belong to the identical population or not.

The hypotheses used in this test are:

H_O: the samples are from identical populations.
H_A the samples are not from identical populations.

The P-value, i.e. the Asymptotic Significance, is provided by SPSS. If it is below 0.05, then the samples are determined not to be from the same population.

3.3 Results of CEM IV / B (P-V) 32.5N

In this part, an inter-laboratory test evaluation performed by TCMA Council for Quality and Environment on compressive strength of a blended cement named as CEM IV / B (P-V) 32.5N is presented.

In this inter-laboratory test evaluation, compressive strength of CEM IV / B (P-V) 32.5N was determined according to TS EN 196-1 in 16 different laboratories. The mix proportion of the mortar is given in Table 3.3.

Table 3.3 The Mix Proportions of CEM IV / B (P-V) 32.5N

Cement Type	**Water to Cement Ratio**	**Ingredients (g)**		
		Cement	**Water**	**Aggregate**
CEM IV B (P-V) 32.5N	0.50	450	225.0	1350

Preparing and casting of all of the mortar specimens were done as follows:

First of all water was poured into the mechanical blender. Then, cement was added to the water and the mixture was mixed for 30 s at low speed. Next, CEN Standard Sand was added and mixed for 30 s. After that, the blend was mixed at high speed for 30 s. Finally, it was left to rest for 90 s and mixed at high speed for 60 s. The prepared mortars were cast into 40 × 40 × 160 mm molds and set in the molds for 24 h. The hardened mortar was then remolded and kept at 20 ± 1 °C in water for 28 day [22].

After 28 day, the specimens were broken into two by flexural strength and the specimens obtained from the flexural strength test were used in compressive strength test. Therefore, the nominal dimensions of the square area subjected to compressive force are 40 × 40 mm. The maximum applied load P on the specimen was determined and the compressive strength R_c was calculated through Equation 3.8.

$$R_c = \frac{P}{A} \tag{3.8}$$

where A is the area subjected to the compressive force, which is equal, in this test, to 1600 mm^2. The load is in Newton and the compressive strength is in N/mm^2.

The standard compressive strength results obtained from 16 laboratories for the pozzolanic cement CEM IV / B (P-V) 32.5N are presented in Table 3.3.

Each laboratory prepared minimum 2 molds. However, two laboratories having the number 10 and 11 were faced with difficulties during the preparation of molds. The mortars were so stiff that they were not effectively compacted into molds. Therefore, these two laboratories tested only one mold.

When coefficients of variances of the test results given in Table 3.4 are examined, it is observed that they vary between 0.78 and 19.78.

Table 3.4 Standard Compressive Strength of CEM IV / B (P-V) 32.5N

Mold Number	Laboratory Number															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	25.4	32.9	24.6	37.3	39.0	37.4	38.2*	27.9	35.0	26.2**	28.0	33.1	19.9*	23.5*	38.3	30.0
	26.0	31.4	24.9	36.8	38.5	39.1	32.0	26.4	35.9	26.8*	29.5	34.0	19.9*	24.5	39.2	28.0
	25.9	28.2*	23.3	37.6	40.6	38.2	37.0	30.1	35.8	37.0*	29.5	33.8	20.8	29.0	38.5	31.0
	26.7	38.5**	26.8	37.1	39.5	38.6	33.2	28.9	36.0	35.4*	31.4	37.0	30.8**	22.1*	39.8	30.0
	25.8	29.4	23.9	37.5	40.7	39.2	29.3**	30.0	36.2	38.5*	29.1	32.1	19.8*	35.0**	40.1	28.7
	26.3	35.5	24.1	37.8	42.5	39.0	36.8	27.8	35.6	36.0	31.3	36.2	25.6*	26.6	40.6	30.7
Min	25.4	28.2	23.3	36.8	38.5	37.4	29.3	26.4	35.0	26.2	28.0	32.1	19.8	22.1	38.3	28.0
Max	26.7	38.5	26.8	37.8	42.5	39.2	38.2	30.1	36.2	38.5	31.4	37.0	30.8	35.0	40.6	31.0
Mean (μ)	26.0	32.7	24.6	37.4	40.1	38.6	34.4	28.5	35.8	33.3	29.8	34.4	22.8	26.8	39.4	29.7
Std. Dev. (σ)	0.44	3.86	1.21	0.36	1.45	0.69	3.47	1.43	0.42	5.39	1.32	1.87	4.51	4.70	0.91	1.16
COV (%)	1.71	11.81	4.93	0.97	3.61	1.78	10.08	5.02	1.17	16.17	4.43	5.44	19.78	17.54	2.31	3.91
2	25.1	33.6*	24.8	36.6	32.0	40.1	32.4	33.2*	31.1			38.9	28.0**	23.6*	40.6	29.0
	26.2	34.0*	25.9	36.4	37.1	39.7	32.8	29.3	31.0			40.1	25.5	25.6	40.1	31.0
	25.7	32.7	25.1	37.0	35.0	38.9	40.3**	25.4*	31.4			32.1**	21.0*	28.3	39.2	30.8
	25.4	33.0	26.0	36.9	36.7	39.6	34.8	32.1	31.9			37.0	21.6	30.3**	39.0	29.6
	26.0	26.3*	25.1	37.0	34.8	37.8	35.6	24.9**	31.6			36.0	27.4*	26.7	40.0	31.6
	26.3	23.2**	27.3	36.4	35.5	40.9	37.7	32.9*	31.9			39.7	20.5*	26.7	39.2	30.0
Min	25.1	23.2	24.8	36.4	32.0	37.8	32.4	24.9	31.0			32.1	20.5	23.6	39.0	29.0
Max	26.3	34.0	27.3	37.0	37.1	40.9	40.3	33.2	31.9			40.1	28.0	30.3	40.6	31.6
Mean (μ)	25.8	30.5	25.7	36.7	35.2	39.5	35.6	29.6	31.5			37.3	24.0	26.9	39.7	30.3
Std. Dev. (σ)	0.47	4.56	0.92	0.29	1.81	1.06	3.01	3.74	0.39			3.00	3.37	2.29	0.64	0.97
COV (%)	1.83	14.96	3.57	0.78	5.15	2.68	8.45	12.62	1.23			8.04	14.05	8.52	1.61	3.19

* denotes the results deviating more than 10% of the mean

** denotes the discarded results

The compressive strength of a single test of the 16 laboratories varies from 19.8 to 42.5 MPa as seen in Figure 3.1. The difference between the minimum and maximum values equal to 22.7 MPa. However, the lower and upper characteristic values given in TS EN 197-1 are 32.5 and 52.5 MPa, i.e. the difference between its minimum and maximum equals to 20 MPa. The interval in which the test results varied is very large when compared with the interval given in TS EN 197-1. There may be several reasons for this situation, such as applicability of the test method, non-homogenous mixing of the materials, water to cement ratio, personel errors, etc.

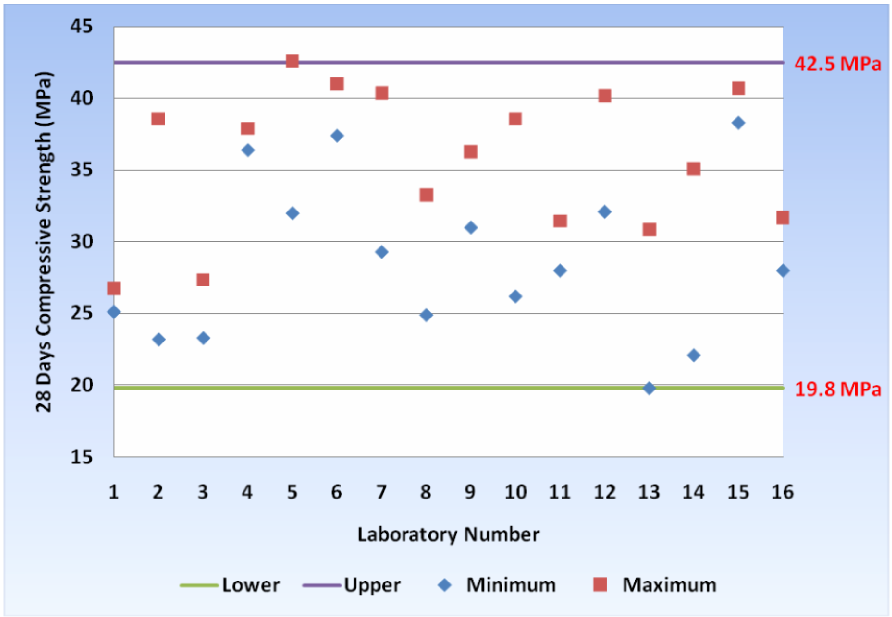


Figure 3.1 The Minimum and Maximum Single Compressive Strength Test Results of Cement Mortar Prepared by CEM IV / B (P-V) 32.5N

The averages of the standard compressive strength obtained by 16 laboratories are given in Figure 3.2. Since the specimen was CEM IV / B (P-V) 32.5N, the minimum and the maximum standard compressive strength must be equal to 32.5 MPa and 52.5 MPa, respectively, according to TS EN 197-1. The results of 8 laboratories did not conform the minimum standard compressive strength characteristic value given in TS EN 197-1.

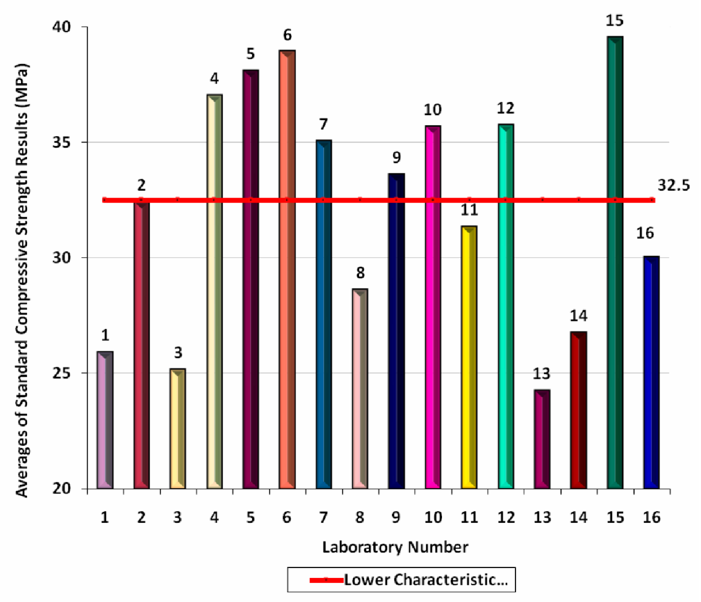


Figure 3.2 Average Compressive Strength of CEM IV / B (P-V) 32.5N as Obtained by Different Laboratories

3.3.1 Acceptance of the Test Results

The test results gathered from 16 laboratories and were examined according to TS EN 196-1. The means of each mold given in Table 3.3 were calculated. Then, the results deviating more than %10 of the mean were detected and also listed in Table 3.3.

For each mold results, one of those values deviating more than 10% of its mean was discarded and the list of these discarded values marked with ** are shown in Table 3.4. After discarding the values marked with **, the means of the rest of the values were calculated. Then, again for the rest of the five results of the molds, the values deviating more than 10% of its recalculated means were determined.

The molds marked as rejected in Table 3.5 still have values deviating more than %10 of the recalculated mean. Thus, the results of the laboratories having the number 2, 10 and 13, the second mold result of the 8th laboratory and the first mold result of the 14th laboratory did not satisfy the requirements of TS EN 196-1. Therefore, they were not included in the evaluation. In other words, total of 7 molds out of 30 were not included in the analysis.

Table 3.5 The List of the Rejected and Accepted Results of CEM IV / B (P-V) 32.5N

Mold Number	Laboratory Number															
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16
1	✓	✕	✓	✓	✓	✓	✓	✓	✓	✕	✓	✓	✕	✕	✓	✓
2	✓	✕	✓	✓	✓	✓	✓	✕	✓			✓	✕	✓	✓	✓

✓ denotes the Accepted test results

✕ denotes the Rejected test results

3.3.2 Omission of Outliers

In this part of the study, the outliers of the results were determined. Before determination of the outliers of the results, it is required to check whether the results are normally distributed. Thus, Kolmogorov-Smirnov Test was performed by using SPSS. It was proved that the test results are normally distributed (see Appendix A).

After confirmation of the normality, outliers of the standard compressive strength results of the 13 laboratories marked as "✓" in Table 3.5 were determined by using Grubb's Test method. In this analysis, the minimum and the maximum values of the test results were determined. The former was 25.2 MPa and the latter was 39.6 MPa. Then, minimum and maximum T values for both of them were calculated by using Equations 3.1 and 3.2 as 1.40 and 1.32, respectively.

These T values were compared with the critical T value of 2.33 given in Table 3.1 for the number of sample 13. Since both of the calculated T values were less than the critical T, there was no outlier in the test results.

Table 3.6 The Means and the Standard Deviations of the Standard Compressive Strength of CEM IV / B (P-V) 32.5N Conforming TS EN 196-1.

Laboratory Number	**Mold Number**						
	1			**2**			
p	**Mean** $\bar{y}_{i1}$	**Standard Deviation** s_{i1}	**Number of Results** n_{i1}	**Mean** $\bar{y}_{i2}$	**Standard Deviation** s_{i2}	**Number of Results** n_{i2}	**Arithmetic Means of Molds**
1	26.0	0.44	6	25.8	0.47	6	25.9
2	-	-	-	-	-	-	-
3	24.6	1.21	6	25.7	0.92	6	25.2
4	37.4	0.36	6	36.7	0.29	6	37.0
5	40.1	1.45	6	35.2	1.81	6	37.7
6	38.6	0.69	6	39.5	1.06	6	39.0
7	35.4	2.68	5	34.7	2.16	5	35.1
8	28.5	1.43	6	-	-	-	-
9	35.8	0.42	6	31.5	0.39	6	33.6
10	-	-	-	-	-	-	-
11	29.8	1.32	6	-	-	-	-
12	34.4	1.77	6	38.3	1.77	5	36.2
13	-	-	-	-	-	-	-
14	-	-	-	26.2	1.73	5	-
15	39.4	0.91	6	39.7	0.64	6	39.6
16	29.7	1.16	6	30.3	0.97	6	30.0
						Min	25.2
						Max	39.6
						Mean	33.9
						Std. Dev.	5.20

3.3.3 Confidence Interval of the Test Results

After estimation of the outliers, for the test results, which were proved to be normally distributed, the 95% confidence interval was determined. In order to determine the interval following steps were performed.

According to the sample size denoted by N, the degree of freedom denoted by N-1 was determined. Then, the t-value corresponding to 2-tailed 95% confidence and the degree of freedom were found out from the t-Table given in Table 3.2. The degree of freedom and t-value used in the analysis were 12 and 2.179, respectively.

By using these values, the 95% confidence interval for the first part of the study was calculated as shown below.

$$\text{Confidence Interval}_{\text{Lower}} = 32.6 - 2.179 \times \frac{5.3}{\sqrt{13}} = 29.5 \qquad \textbf{(3.11)}$$

$$\text{Confidence Interval}_{\text{Upper}} = 32.6 + 2.179 \times \frac{5.3}{\sqrt{13}} = 35.8 \qquad \textbf{(3.12)}$$

According to findings of the evaluation, Figure 3.3 was plotted. The standard compressive strength test results of the 13 laboratories included in the analysis and the mean of them are given in this figure. In addition, the 95% confidence interval of the sample is shown.

It is observed that only 4 laboratories out of 13, i.e. 31%of the laboratories, fall into the 95% confidence interval. The results reveal that the bulk of the laboratories are out of the 95% confidence interval.

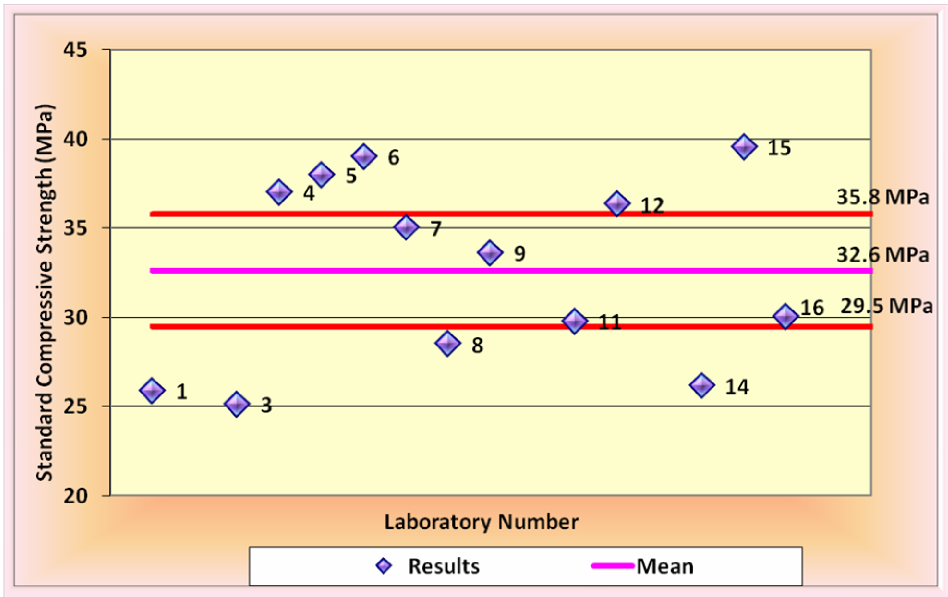


Figure 3.3 The Means of the Standard Compressive Strength of the Laboratories Included in the Analysis

3.3.4 Calculation of the Repeatability and Reproducibility

The standard deviation of the repeatability and the reproducibility of the test results were calculated by Equations 3.4 and 3.7, respectively, for each mold numbers separately.

As a result, for the repeatability standard deviation and reproducibility standard deviation was calculated as s_r = 2.50 and s_R = 5.61, respectively. These values are presented in Table 3.7.

Table 3.7 The Repeatability and the Reproducibility of CEM IV / B (P-V) 32.5N

Mold Number *j*	Number of Laboratories *p*	s_{rj}	s_{Rj}	Repeatability		Reproducibility	
1	16	2.64	5.64	Std. Dev.	COV	Std. Dev.	COV
2	14	2.34	5.57	2.50	7.80*	5.61	17.50*

* Outside the acceptable limits

The coefficient of variance of the repeatability is expected to be in between 1 and 3 % according to TS EN 196-1. In addition, the coefficient of variance of the reproducibility is expected to be less than 6%. However, the repeatability and reproducibility coefficient of variances are much more higher than the expected values given in TS EN 196-1.

3.3.5 Comparison According to Mann-Whitney Test

In addition to inter-laboratory test comparison performed, the results were also examined in SPSS by using Mann-Whitney Test method. In this test method, whether the test results obtained belong to the same population or not are examined. The results of the Mann-Whitney Test method applied to the first part of the study are given in Table 3.8.

If those test results were belong to the same population, then the asymptotic significance values are expected to more than 0.05. In Table 3.8, the asymptotic significance values more than 0.05 are denoted by "✓", whereas less than 0.05

are denoted by "✗". It is clear that most of the samples do not belong to the same population. Although the same test method was applied to the initial specimens, the results were not similar.

Table 3.8 The Comparison of CEM IV / B (P-V) 32.5N Results

Laboratory Number

2	✗														
3	✗	✗													
4	✗	✗	✗												
5	✗	✗	✗	✓											
6	✗	✗	✗	✗	✓										
7	✗	✓	✗	✓	✗	✗									
8	✗	✓	✗	✗	✗	✗	✗								
9	✗	✓	✗	✗	✗	✗	✓	✗							
10	✗	✓	✗	✓	✗	✗	✓	✓	✓						
11	✗	✓	✗	✗	✗	✗	✗	✓	✗	✓					
12	✗	✗	✗	✓	✗	✗	✓	✗	✗	✓	✗				
13	✓	✗	✓	✗	✗	✗	✗	✗	✗	✗	✗	✗			
14	✓	✗	✓	✗	✗	✗	✗	✓	✗	✗	✗	✗	✗		
15	✗	✗	✗	✗	✓	✓	✗	✗	✗	✗	✗	✗	✗	✗	
16	✗	✓	✗	✗	✗	✗	✗	✓	✗	✓	✓	✗	✗	✗	✗
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15

Laboratory Number

According to the results of Inter-laboratory Test and Mann-Whitney Test, it is concluded that the test method applied for the blended cement CEM IV / B (P-V) 32.5N was not appropriate.

CHAPTER 4

EXPERIMENTAL PROGRAM

4.1 General

According to results gathered from the inter-laboratory test obtained by TCMA – Council for Quality and Environment, it is decided to perform the compressive strength test on three cement types; namely, CEM I 42.5R , CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R. However, the organization of the tests is performed by Materials of Construction Laboratory at Middle East Technical University (METU). Thus, the size of the participating laboratories is very limited.

4.2 Materials

The cement types used in all mixtures prepared in the second part of the study were labeled as CEM I 42.5R, CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R according to TS EN 197-1. The cements were obtained from the local market in tetrabags of 50 kg.

The aggregate used in the study was CEN Standard Sand conforming TS EN 196 – 1 obtained from the local market.

4.3 Mixture preparation

Two different cement mortar mixes were prepared for each type of cements. In the first mix, water to cement ratio of the cement pastes were the same as given in TS EN 196-1. In the second mix, flow diameter of the cement mixes were fixed at 16 cm. The compressive strength of the mortar mixes were tested in five different laboratories. The mix proportions of these cement mortars are given in Table 4.1.

Table 4.1 Mix Proportions of CEM I 42.5R, CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R

Cement Type	Mortar Mix	Water to Cement Ratio	Ingredients (g)			Flow (cm)
			Cement	Water	Aggregate	
CEM I 42.5R	Constant Water Content	0.50	450	225.0	1350	15
	Constant Flow	0.49	500	242.5	1375	16
CEM II / A-M (P-S) 42.5R	Constant Water Content	0.50	450	225.0	1350	14
	Constant Flow	0.50	500	250.0	1375	16
CEM IV B (P) 32.5R	Constant Water Content	0.50	450	225.0	1350	12
	Constant Flow	0.54	500	276.5	1375	16

4.4 Chemical composition of cements

The cement is composed of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO as major oxides and K_2O, Na_2O, TiO_2, P_2O_5 and SO_3 as minor oxides. In the second part of the study, determination of the oxide composition was done by X-Ray fluorescence (XRF).

4.5 Fineness of cements

4.5.1 Specific Surface – Blaine Fineness

The fineness is determined from the air permeability of a bed of cement of specified porosity using the Blaine air permeability apparatus and expressed as the surface area in square centimeters per gram (cm^2/g) of cement according to the expression given below.

$$S = \frac{A\sqrt{t}}{d} \tag{4.1}$$

where S is the Blaine Fineness in cm^2/g, A is the coefficient of the apparatus which is determined by calibration, t is the time elapsed to draw the air through the cement bed and d is the density of the cement.

4.5.2 Particle Size Distribution (PSD)

Particle size distribution is another method to determine the fineness of the cement. The laser technique is used for the determination of the particle size distribution.

4.5.3 Sieve Analysis

Another technique to determine the fineness of the cement is to sieve it through certain size mash sieves. This analysis is carried out according to TS EN 196-6.

4.6 Tests on Mortars

4.6.1 Slump Flow Diameter

The test method used for the determination of the slump flow for a cement mortar was ASTM C 1437. For this test, a flow mold was used. The flow mold was placed at the center of the flow table, then the mold was filled with the mortar till 25 mm and then mortar was tampered 20 times. After tampering, the rest of the mold was again filled with the mortar and tampered as described for the previous layer. Then, the upper surface of the mold was flattened by trowel and the mold was carefully removed. Immediately the mortar was dropped 25 times in 15 s to spread. After spreading finished, two perpendicular diameters were measured and their average diameter was taken as slump flow diameter [23].

4.6.2 Compressive Strength Test

The compressive strength was determined according to TS EN 196 – 1. The nominal dimensions of the square area subjected to compressive force in the specimens are 40 × 40 mm. The maximum applied load P on the specimen was recorded and the compressive strength R_c was calculated using the expression below.

$$R_c = \frac{P}{A} \qquad \textbf{(4.2)}$$

where A is the area subjected to the compressive force, which is equal, in this case, to 1600 mm^2. The load is in N and the compressive strength is in N/mm^2.

CHAPTER 5

EXPERIMENTAL RESULTS

5.1 General

In this part of the study, three different types of cements namely, CEM I 42.5R, CEM II / A-M (P-S) 42.5R and CEM IV / B (P) 32.5R were used. For each type of cement, two different cement mortars were prepared and compressive strength of these mortars were tested at 2 and 28 day.

5.2 CEM I 42.5R

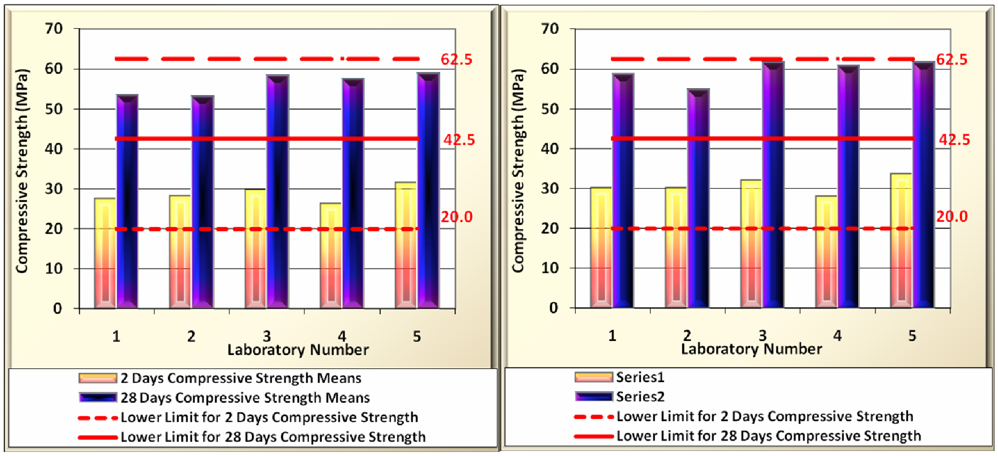
The results of 2 and 28 day compressive strength of cement mortar mixes having constant water content and constant flow prepared with CEM I 42.5R were given in Table 5.1.

The results of constant water content mortar for 2 day compressive strength vary in between 25.1 and 31.9 MPa. The results for 28 day compressive strength vary in between 49.1 and 60.0 MPa. The results of constant flow mortar for 2 day compressive strength vary in between 22.3 and 33.8 MPa, while those for 28 day compressive strength vary in between 50.3 and 63.4 MPa. It is obvious that the interval of the results obtained by constant water content is narrower than those obtained by constant flow for 2 and 28 day compressive strength.

Table 5.1 Compressive Strength Results of CEM I 42.5R

Compressive Strength	Constant water content						Constant flow					
	Laboratory Number						Laboratory Number					
	1		2	3	4	5	1		2	3	4	5
	Mold#1	Mold#2	Mold#1	Mold#1	Mold#1	Mold#1	Mold#1	Mold#2	Mold#1	Mold#1	Mold#1	Mold#1
2-Day	27.0	27.0	27.0	30.4	26.2	30.8	23.2*	33.2	28.8	32.1	27.9	33.7
	26.7	26.4	27.8	30.0	25.1	31.8	32.2*	31.6	29.3	31.8	28.2	33.8
	28.1	28.4	28.3	29.2	27.2	31.9	22.3*	30.8	30.1	32.3	27.3	33.8
	26.8	29.7	27.3	30.1	26.4	31.7	31.4	30.6	29.0	32.4	27.1	33.4
	27.6	27.4	29.1	29.2	27.0	31.5	30.8	30.9	31.4	31.6	28.6	33.8
	27.4	27.1	28.8	29.4	25.8	31.3	32.7*	30.9	31.7	32.0	28.2	32.5
Min	**26.7**	**26.4**	**27.0**	**29.2**	**25.1**	**30.8**	**22.3**	**30.6**	**28.8**	**31.6**	**27.1**	**32.5**
Max	**28.1**	**29.7**	**29.1**	**30.4**	**27.2**	**31.9**	**32.7**	**33.2**	**31.7**	**32.4**	**28.6**	**33.8**
Mean	**27.3**	**27.7**	**28.1**	**29.7**	**26.3**	**31.5**	**28.1**	**31.3**	**30.1**	**32.0**	**27.9**	**33.5**
St.Dev.	**0.54**	**1.19**	**0.83**	**0.52**	**0.78**	**0.40**	**4.92**	**0.98**	**1.25**	**0.30**	**0.58**	**0.51**
COV (%)	**1.96**	**4.31**	**2.96**	**1.73**	**2.95**	**1.29**	**17.54**	**3.11**	**4.15**	**0.94**	**2.07**	**1.53**
28-Day	52.6	54.6	52.3	58.4	57.6	58.1	63.1	58.2	54.0	63.4	61.0	61.8
	56.0	54.5	53.9	59.0	56.3	57.8	59.5	60.0	56.3	62.3	59.2	61.7
	53.3	54.3	54.0	57.5	57.2	59.3	60.2	55.5	53.9	61.0	62.0	62.2
	49.4	54.9	52.7	57.4	57.6	57.7	60.0	60.3	54.6	61.4	60.6	61.0
	49.1	52.6	52.1	58.1	58.4	60.0	50.3*	58.3	55.2	60.4	61.1	62.2
	51.9	56.1	53.1	58.6	57.2	59.1	57.0	59.6	54.1	61.0	60.4	60.9
Min	**49.1**	**52.6**	**52.1**	**57.4**	**56.3**	**57.7**	**50.3**	**55.5**	**53.9**	**60.4**	**59.2**	**60.9**
Max	**56.0**	**56.1**	**54.0**	**59.0**	**58.4**	**60.0**	**63.1**	**60.3**	**56.3**	**63.4**	**62.0**	**62.2**
Mean	**52.1**	**54.5**	**53.0**	**58.2**	**57.4**	**58.7**	**58.4**	**58.7**	**54.7**	**61.6**	**60.7**	**61.6**
St.Dev.	**2.58**	**1.13**	**0.80**	**0.63**	**0.69**	**0.94**	**4.40**	**1.77**	**0.93**	**1.09**	**0.93**	**0.57**
COV (%)	**4.95**	**2.07**	**1.51**	**1.08**	**1.20**	**1.59**	**7.54**	**3.02**	**1.70**	**1.77**	**1.53**	**0.92**

* denotes the results deviating more than 10% of the mean



(a) Constant Water Content **(b)** Constant Flow

Figure 5.1 The Average Compressive Strength of CEM I 42.5R

The early compressive strength requirement defined in TS EN 197-1 for CEM I 42.5R is 20.0 MPa. The lower and the upper standard compressive strength requirements defined in TS EN 197-1 are 42.5 and 62.5MPa, respectively. It is obvious that the averages of the early and standard compressive strengths of the cement mortar mixes constant water content and constant flow satisfy the requirements of TS EN 197-1.

5.2.1 Acceptance of the Results

For cement mortar mixes having constant water content and constant flow, 2 and 28 day compressive strength results were compared with their means.

When results of constant water content mortar were compared, it was recognized that there was no result deviating more than 10% of its mean. Thus, all of the results obtained for cement mortar mix having constant water content were accepted.

When results of constant flow mortar were compared, it is observed that 2 and 28 day compressive strength results of the laboratory number 1 marked with * in Table 5.1 deviated more than 10% of its mean. However, in order to observe the effect of these results, they were not discarded.

5.2.2 Omission of Outliers

First, the test results were examined by Kolmogorov-Smirnov Test to see if they are normally distributed. The test result revealed that data sets are normally distributed (see Appendix A).

Then, determination of outliers is performed. It is observed that for 28 day compressive strength of constant flow mortar, there is an outlier belonging to the laboratory number 2. However, as the number of participating laboratories is rather small, it was not omitted.

5.2.3 Confidence Interval of the Test Results

According to the sample size, the degrees of freedoms are four both for constant water content and constant flow mortars. Then, the corresponding t value obtained from Table 3.2 is 2.776.

Finally, the 95% confidence interval for constant water content and constant flow was obtained.

There is one 2 day compressive strength result out of the limits for cement mortar mixes obtained by CEM I 42.5R for both constant water content and constant flow as seen in Figure 5.2. Thus, these results are out of the confidence interval. It is observed that there is no big difference between 2 day compressive strength of constant water content and constant flow. However, for 28 day compressive strength, the confidence interval of constant water content mortar is bigger than the confidence interval of constant flow mortar. In addition, there is an outlier in the results of constant flow.

Moreover, the results obtained for constant flow mortar is approximately three MPa higher than those obtained for constant water content mortar for both constant water content and constant flow. The constant water content results are more reliable than constant flow results.

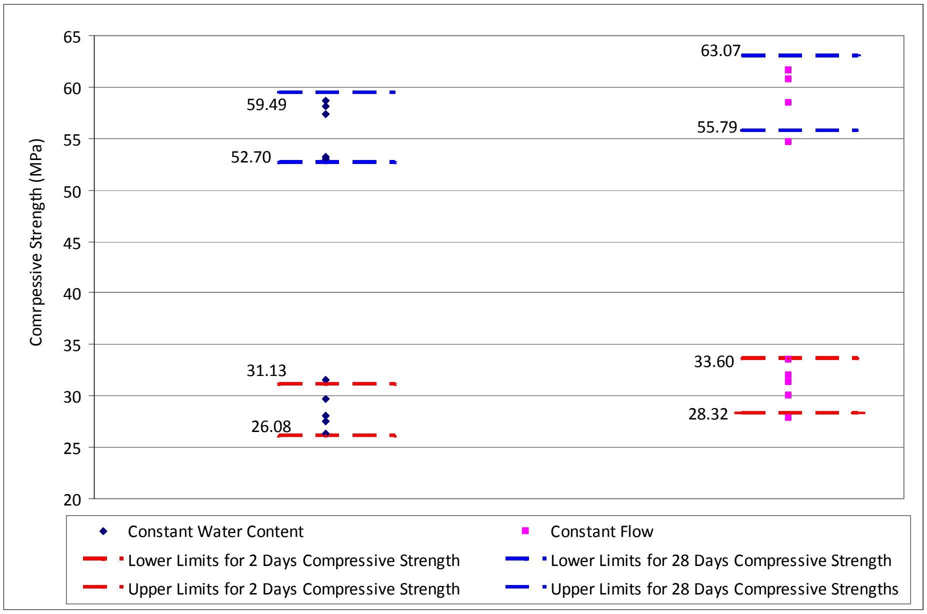


Figure 5.2 95% Confidence Interval for CEM I 42.5R

5.2.4 Calculation of the Repeatability and Reproducibility

The repeatability and the reproducibility were calculated according to Equations 3.4 and 3.7 and are given in Table 5.2.

When coefficient of variance of the repeatability and reproducibility are compared with the repeatability and reproducibility limits defined in TS EN 196-1, it is observed that the repeatabilities belonging to the constant water content mortars are in the limits for both 2 and 28 day compressive strength. Whereas, the repeatabilities belonging to the constant flow mortars are outside the limit for both 2 and 28 day compressive strength.

Table 5.2 Standard Deviation and COV of the Repeatability and the Reproducibility.

	Compressive Strength (Days)	Mold Number *j*	Number of Laboratories *p*	s_{rj}	s_{Rj}	Repeatability		Reproducibility	
						Std. Dev.	COV	Std. Dev.	COV
Constant Water Content	2	1	5	0.6	1.9	0.7	2.5	1.8	6.2*
		2	1	1.2	1.2				
	28	1	5	1.3	2.9	1.3	2.3	2.6	4.7
		2	1	1.1	1.1				
Constant Flow	2	1	5	2.2	2.6	2.0	6.5*	2.3	7.6*
		2	1	1.0	1.0				
	28	1	5	2.1	3.0	2.1	3.5	2.8	4.8
		2	1	1.8	1.8				

* Outside the acceptable limits

However, for both constant water content and constant flow mortars the coefficient of variance of the reproducibilities for 2 day compressive strength are higher than 6% as defined in TS EN 196-1. Whereas, for 28 day compressive strength both reproducibilities belonging to the constant water content and constant flow mortars are within the limits.

Thus, the findings revealed that the results of constant water content are more reliable than those of constant flow.

5.2.5 Comparison According to Mann-Whitney Test

As seen in Table 5.3, 2 and 28 day compressive strengths of CEM I 42.5R, there is not any difference in between TS EN 196-1 and constant flow test method.

Table 5.3 The Comparison of CEM I 42.5R Results

Compressive Strength (Days)	2								28							
Mixes	Constant Water Content				Constant Flow				Constant Water Content				Constant Flow			
2	✓				✓				✓				X			
3	X	X			✓	X			X	X			X	X		
4	✓	X	X		X	X	X		X	X	X		X	X	✓	
5	X	X	X	X	X	X	X	X	X	X	✓	X	X	X	✓	✓
Laboratory Number	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4

✓denotes similar populations
X denotes different populations

5.3 CEM II / A-M (P-S) 42.5R

Compressive strength of cement mortar mixes having constant water content and constant flow, and prepared with cement CEM II / A-M (P-S) 42.5R are presented.

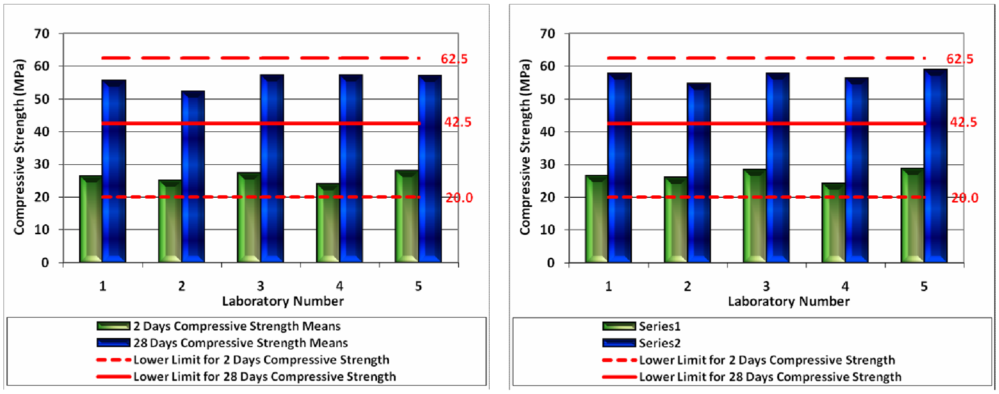
The results of 2 and 28 day compressive strength obtained for constant water content mortar mix that had mix proportions as defined in TS EN 196-1 are given in Table 5.4. The results of this mortar for 2 day compressive strength varies in between 23.6 and 28.4 MPa. Whereas, the results of this mortar for 28 day compressive strength vary in between 51.3 and 60.1 MPa.

The results of 2 and 28 day compressive strength obtained for constant flow mortar are also given in Table 5.4. The results of this cement mortar mix vary in between 22.9 and 29.2 MPa for 2 day compressive strength, while those for 28 day compressive strength vary in between 53.1 and 60.5 MPa.

It is obvious that the interval of the results obtained for constant water content mortar is narrower than those obtained for constant flow mortar for 2 day compressive strength. While the interval of the test results obtained for constant flow mortar is narrower than those results obtained for constant water content for 28 day compressive strength.

Table 5.4 Compressive Strength Results of CEM II / A-M (P-S) 42.5R

Compressive Strength	**Constant water content**						**Constant flow**					
	Laboratory Number						**Laboratory Number**					
	1		**2**	**3**	**4**	**5**	**1**		**2**	**3**	**4**	**5**
	Mold#1	**Mold#2**	**Mold#1**	**Mold#1**	**Mold#1**	**Mold#1**	**Mold#1**	**Mold#2**	**Mold#1**	**Mold#1**	**Mold#1**	**Mold#1**
2-Day	26.2	27.6	24.2	27.3	23.6	27.8	26.0	26.4	25.4	27.5	24.8	28.4
	25.3	26.6	25.5	27.8	23.9	28.0	25.7	27.6	26.2	28.7	23.8	28.6
	26.4	25.3	26.1	27.8	23.6	27.6	27.3	26.7	26.9	28.9	23.9	28.8
	26.6	25.4	24.6	27.4	24.3	28.4	27.8	26.8	25.6	27.6	22.9	29.2
	26.0	26.5	25.2	26.3	24.7	27.7	26.2	24.3	26.4	28.9	25.6	28.7
	26.1	27.9	24.1	26.6	23.9	28.3	27.8	25.5	25.2	28.6	23.6	28.5
Min	**25.3**	**25.3**	**24.1**	**26.3**	**23.6**	**27.6**	**25.7**	**24.3**	**25.2**	**27.5**	**22.9**	**28.4**
Max	**26.6**	**27.9**	**26.1**	**27.8**	**24.7**	**28.4**	**27.8**	**27.6**	**26.9**	**28.9**	**25.6**	**29.2**
Mean	**26.1**	**26.6**	**25.0**	**27.2**	**24.0**	**28.0**	**26.8**	**26.2**	**26.0**	**28.4**	**24.1**	**28.7**
St.Dev.	**0.45**	**1.08**	**0.79**	**0.62**	**0.43**	**0.33**	**0.94**	**1.16**	**0.66**	**0.64**	**0.95**	**0.28**
COV (%)	**1.71**	**4.06**	**3.15**	**2.29**	**1.79**	**1.17**	**3.52**	**4.42**	**2.53**	**2.27**	**3.96**	**0.99**
28-Day	57.4	58.1	51.3	58.1	55.9	58.4	56.9	57.7	54.4	55.9	57.7	60.5
	55.6	57.0	53.1	57.4	55.8	57.9	58.9	56.1	55.3	58.4	56.2	59.7
	54.7	56.2	52.1	56.5	60.1	56.0	55.9	57.6	54.9	58.3	54.8	59.5
	57.4	54.7	51.7	55.7	55.7	56.1	55.8	58.3	54.6	59.1	57.1	59.9
	54.1	55.1	53.2	57.3	57.3	57.6	58.7	59.7	55.4	58.4	55.2	56.2
	53.8	53.2	52.1	58.7	58.7	56.8	59.3	58.5	53.1	56.7	56.0	57.3
Min	**53.8**	**53.2**	**51.3**	**55.7**	**55.7**	**56.0**	**55.8**	**56.1**	**53.1**	**55.9**	**54.8**	**56.2**
Max	**57.4**	**58.1**	**53.2**	**58.7**	**60.1**	**58.4**	**59.3**	**59.7**	**55.4**	**59.1**	**57.7**	**60.5**
Mean	**55.5**	**55.7**	**52.3**	**57.3**	**57.3**	**57.1**	**57.6**	**58.0**	**54.6**	**57.8**	**56.2**	**58.9**
St.Dev.	**1.59**	**1.75**	**0.76**	**1.08**	**1.82**	**0.99**	**1.58**	**1.19**	**0.84**	**1.22**	**1.10**	**1.70**
COV (%)	**2.87**	**3.14**	**1.45**	**1.88**	**3.18**	**1.73**	**2.74**	**2.05**	**1.53**	**2.12**	**1.96**	**2.88**



(a) Constant Water Content **(b)** Constant Flow

Figure 5.3 The Average Compressive Strength of CEM II / A-M (P-S) 42.5R

The early compressive strength requirement defined in TS EN 197-1 for CEM II / A-M (P-S) 42.5R is 20.0 MPa. The lower and the upper standard compressive strength requirements are 42.5 MPa and 62.5 MPa, respectively. Then, it is clear that the averages of the early and standard compressive strength of the cement mortar mixes obtained by CEM II / A-M (P-S) 42.5R satisfy the requirements of TS EN 197-1.

5.3.1 Acceptance of the Results

When a comparison is made between the results of 2 and 28 day compressive strength of cement mortar mixes prepared by CEM II / A-M (P-S) 42.5R and their means, it was recognized that there was no result deviating more than 10% of its mean. Thus, all of the data satisfied the requirements defined in TS EN 196-1.

5.3.2 Omission of Outliers

The test results examined by Kolmogorov-Smirnov Test revealed that they are normally distributed. According to this test result data sets are normally distributed (see Appendix A).

Then, determination of outliers is performed. It is observed that there is an outlier belonging to laboratory number 2 for 28 day compressive strength of constant water content mortar. However, again as the number of laboratories is rather small, the test result of the laboratory number 2 is not omitted.

5.3.3 Confidence Interval of the Test Results

According to the sample size the degrees of freedom are four for constant water content and constant flow mortars. Then, the corresponding t value obtained from Table 3.2 is 2.776.

Finally, the 95% confidence interval both for constant water content and constant flow is obtained and given in Figure 5.4. For 2 day compressive strength of cement mortar having constant flow, it is observed that there is a result out of the confidence interval. However, for constant water content, there is not any result out of the confidence interval.

In addition, there is a result out of the confidence interval for 28 day compressive strength for both cement mortar having constant water content and constant flow.

When the confidence intervals of the cement mortar mixes obtained for constant water content and constant flow are compared, it is obvious that the interval of constant water content mortar is similar to the interval of constant flow mortar.

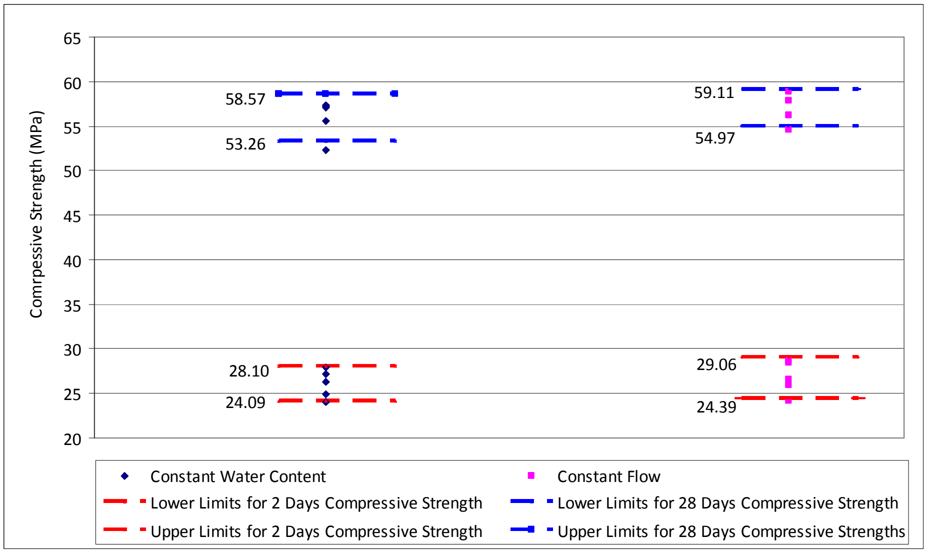


Figure 5.4 95% Confidence Interval for CEM II / A-M (P-S) 42.5R

5.3.4 Calculation of the Repeatability and Reproducibility

The repeatability and the reproducibility were calculated by Equations 3.4 and 3.7 and given in Table 5.5.

When coefficient of variance of the repeatability and reproducibility are compared with the repeatability and reproducibility limits defined in TS EN 196-1, it is observed that the repeatabilities and reproducibilites belonging to the constant water content mortars are in the limits for both 2 and 28 day compressive strength. Whereas, the repeatabilities belonging to the constant flow mortars are in the limits for both 2 and 28 day compressive strength.

Table 5.5 The Repeatability and the Reproducibility of CEM II / A-M (P-S) 42.5R

	Compressive Strength (Days)	Mold Number *j*	Number of Laboratories *p*	s_{rj}	s_{Rj}	Repeatability		Reproducibility	
						Std. Dev.	COV	Std. Dev.	COV
Constant Water Content	2	1	5	0.5	1.5	0.6	2.4	1.4	5.5
		2	1	1.1	1.1				
	28	1	5	1.3	2.1	1.4	2.5	2.1	3.7
		2	1	1.7	1.7				
Constant Flow	2	1	5	0.7	1.8	0.8	3.0	1.7	6.2*
		2	1	1.2	1.2				
	28	1	5	1.3	1.8	1.3	2.3	1.7	2.9
		2	1	1.2	1.2				

* Outside the acceptable limits

However, for constant flow mortars the coefficient of variance of the reproducibility for 2 day compressive strength is higher than 6% that defined in TS EN 196-1. Whereas, for 28 day compressive strength, the reproducibility belonging to the constant flow mortar is in the limit.

It is obvious that there is not much difference in between constant water content and constant flow for CEM II / A-M (P-S) 42.5R.

5.3.5 Comparison According to Mann-Whitney Test

The results of the Mann-Whitney Test are summarized in Table 5.6. For 2 and 28 day compressive strengths of CEM II / A-M (P-S) 42.5R, the test results obtained for constant flow test method has given more identical test results when compared to TS EN 196-1 test method.

Table 5.6 The Comparison of CEM II / A-M (P-S) 42.5R Results

Mixes	Compressive Strength (Day)															
	2								28							
	Constant Water Content				Constant Flow				Constant Water Content				Constant Flow			
2	X				✓				X				X			
3	X	X			X	X			X	X			✓	X		
4	X	X	X		X	X	X		X	X	X		X	X	✓	
5	X	X	X	X	X	X	✓	X	X	X	X	X	✓	X	✓	X
Laboratory Number	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4

✓denotes similar populations

X denotes different populations

5.4 CEM IV / B (P) 32.5R

The results of 2 and 28 day compressive strength obtained for constant water content mortar that had the same mix proportions as defined in TS EN 196-1 are given in Table 5.7. The results of this mortar mix for 2 day compressive strength vary in between 9.9 and 14.5 MPa while those for 28 day compressive strength vary in between 34.5 and 41.2 MPa.

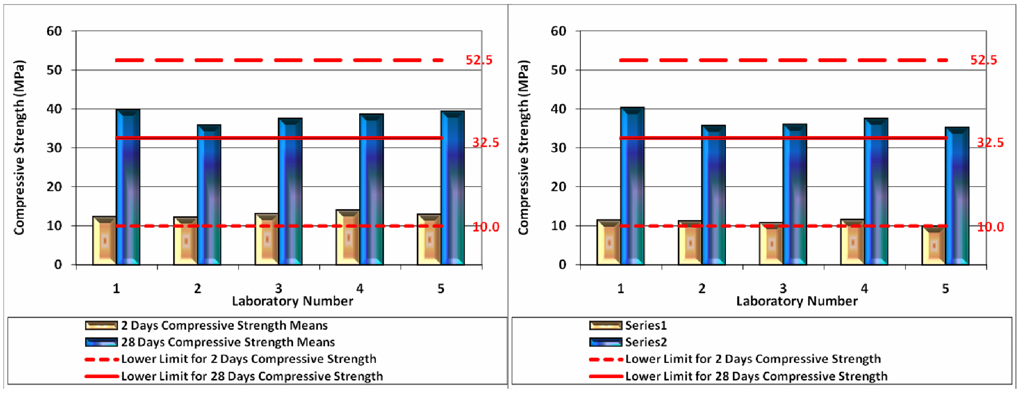
In addition, the results of 2 and 28 day compressive strength obtained for constant flow mortar are also given in Table 5.7. The results for 2 day compressive strength vary in between 9.9 and 12.8 MPa, while those for 28 day compressive strength vary in between 34.1 and 41.5 MPa.

It is obvious that the interval of the results obtained for constant flow mortar is narrower than those obtained for constant water content mortar for 2 day compressive strength. While the interval of the test results obtained for constant water content is similar to those obtained for constant flow mortar for 28 day compressive strength.

Table 5.7 Compressive Strength of CEM IV / B (P) 32.5R

Compressive Strength	Constant water content						Constant flow					
	Laboratory Number						Laboratory Number					
	1		2	3	4	5	1		2	3	4	5
	Mold#1	Mold#2	Mold#1	Mold#1	Mold#1	Mold#1	Mold#1	Mold#2	Mold#1	Mold#1	Mold#1	Mold#1
2-Day	14.0	9.9*	12.4	13.4	12.5*	12.8	12.3	10.6	9.9*	10.8	11.1	9.9
	13.9	10.6	13.0	12.9	14.5	13.0	12.3	10.7	11.0	10.7	10.8	9.8
	13.3	10.1*	13.0	12.8	14.4	12.9	11.9	10.6	10.7	10.6	11.3	10.0
	13.8	10.9	10.2*	13.6	13.7	13.2	12.1	10.9	12.0	10.5	12.8*	9.7
	10.4*	13.7*	13.5*	13.1	14.3	12.8	12.2	10.3	12.3*	11.2	11.8	9.9
	11.7	14.3*	10.7*	13.0	14.1	12.8	11.9	11.0	11.1	10.8	11.2	9.6
Min	**10.4**	**10.1**	**10.2**	**12.8**	**12.5**	**12.8**	**11.9**	**10.3**	**9.9**	**10.5**	**10.8**	**9.6**
Max	**14.0**	**14.3**	**13.5**	**13.6**	**14.5**	**13.2**	**12.3**	**11.0**	**12.3**	**11.2**	**12.8**	**10.0**
Mean	**12.9**	**11.9**	**12.1**	**13.1**	**13.9**	**12.9**	**12.1**	**10.7**	**11.2**	**10.8**	**11.5**	**9.8**
St.Dev.	**1.47**	**1.93**	**1.36**	**0.31**	**0.75**	**0.16**	**0.18**	**0.25**	**0.88**	**0.24**	**0.72**	**0.15**
COV (%)	**11.47**	**16.21**	**11.20**	**2.34**	**5.39**	**1.24**	**1.51**	**2.32**	**7.84**	**2.25**	**6.22**	**1.50**
28-Day	38.6	39.4	34.5	36.2	39.0	39.7	39.1	38.9	34.1	35.5	37.1	35.5
	39.8	41.1	35.1	37.2	38.3	39.2	40.3	40.3	35.6	35.9	36.2	34.9
	38.8	39.4	36.8	38.8	39.5	39.3	39.9	39.6	36.8	37.2	37.2	35.9
	39.4	40.6	35.2	37.0	37.8	39.2	39.6	39.8	36.1	35.2	39.2	35.6
	37.6	40.8	37.1	37.7	38.1	39.4	40.7	41.4	35.7	36.1	38.1	34.3
	40.6	41.2	36.3	37.4	38.5	38.8	41.1	41.5	35.2	36.0	36.9	34.6
Min	**37.6**	**39.4**	**34.5**	**36.2**	**37.8**	**38.8**	**39.1**	**38.9**	**34.1**	**35.2**	**36.2**	**34.3**
Max	**40.6**	**41.2**	**37.1**	**38.8**	**39.5**	**39.7**	**41.1**	**41.5**	**36.8**	**37.2**	**39.2**	**35.9**
Mean	**39.1**	**40.4**	**35.8**	**37.4**	**38.5**	**39.3**	**40.1**	**40.3**	**35.6**	**36.0**	**37.5**	**35.1**
St.Dev.	**1.04**	**0.82**	**1.05**	**0.86**	**0.62**	**0.29**	**0.73**	**1.03**	**0.91**	**0.69**	**1.05**	**0.63**
COV (%)	**2.66**	**2.02**	**2.92**	**2.30**	**1.61**	**0.75**	**1.83**	**2.57**	**2.55**	**1.90**	**2.81**	**1.79**

* denotes the results deviating more than 10% of the mean



(a) Constant Water Content **(b)** Constant Flow

Figure 5.5 The Average Compressive Strength of CEM IV / B (P) 32.5R

The early compressive strength requirement defined in TS EN 197-1 for CEM IV / B (P) 32.5R is 10.0 MPa. The lower and the upper standard compressive strength requirements defined in TS EN 197-1 are 32.5 MPa and 52.5 MPa, respectively. Then, it is clear that the averages of each laboratory for early and standard compressive strength of the cement mortar mixes having constant water content satisfy the requirements of TS EN 197-1. In addition, the average compressive strength of cement mortar mixes having constant flow satisfy the requirements defined in TS EN 197-1 in most of the laboratories, except in laboratory 5.

5.4.1 Acceptance of the Results

When a comparison is made between the results of CEM IV / B (P) 32.5R and their means, it is obvious that there are several 2 day compressive strength results deviating more than 10% of its mean marked with "*" in Table 5.7. There are nine compressive strength results outside the limits of TS EN 196-1 for mortar mix having constant water content, whereas the number of outside results is 3 for mortar mix having constant flow.

Although, TS EN 196-1 states that the result deviating more than 10% of its mean should be discarded, in order to observe the effects of those results none of them was discarded.

When a comparison of the results mentioned above is made, it is clear that the number of results outside the requirements defined in TS EN 196-1 is much more higher for CEM IV / B (P) 32.5R than the other cements.

5.4.2 Omission of Outliers

The test results examined by Kolmogorov-Smirnov Test revealed that they are normally distributed. According to this test result, data sets are normally distributed (see Appendix A).

Then, when the test results are compared with the critical T values obtained from Table 3.1, it is observed that there is no outlier both for constant water content and constant flow mortars prepared with CEM IV / B (P) 32.5R.

5.4.3 Confidence Interval of the Test Results

Since there are five results for both constant water content and constant flow, the degrees of freedom equals to four and the corresponding t value is 2.776. The 95% confidence interval both for constant water content and constant flow was obtained and given in Figure 5.6.

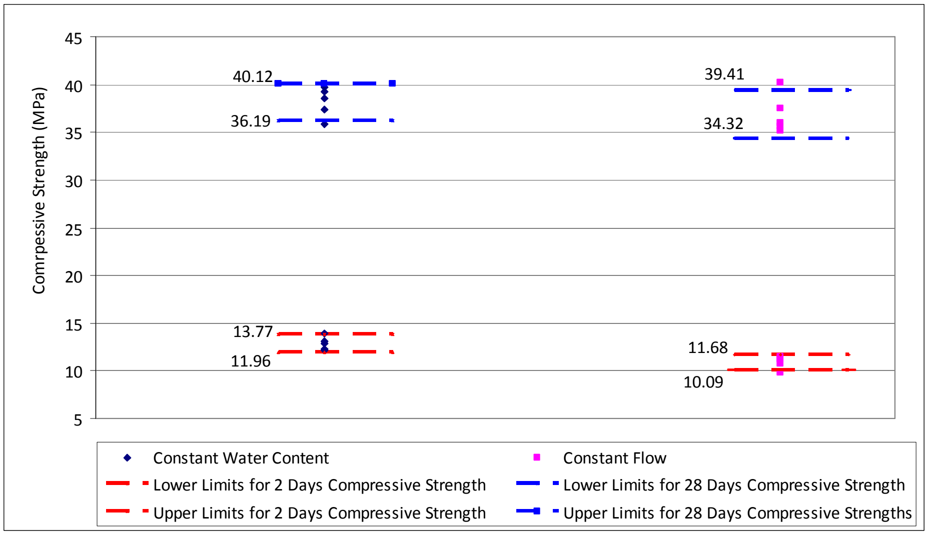


Figure 5.6 95% Confidence Interval for CEM IV / B (P) 32.5R

When the confidence intervals of constant water content and constant flow are compared, it is obvious that the interval of constant flow is narrower than constant water content for 2 day compressive strength. However, for 28 day compressive strength, constant water content confidence interval is narrower than the interval of constant flow.

Constant water content has results out of the confidence interval both for 2 and 28 day compressive strength, whereas, constant flow has a result out of the confidence interval only for 28 day compressive strength. If the results outside the confidence intervals of both constant water content and constant flow were omitted, the interval of constant flow would be much more narrower than constant water content.

5.4.4 Calculation of the Repeatability and Reproducibility

The repeatability and the reproducibility were calculated by Equations 3.4 and 3.7 and given in Table 5.8.

Table 5.8 The Repeatability and the Reproducibility of CEM IV / B (P) 32.5R

	Compressive Strength (Days)	Mold Number *j*	Number of Laboratories *p*	s_{rj}	s_{Rj}	Repeatability		Reproducibility	
						Std. Dev.	COV	Std. Dev.	COV
Constant Water Content	2	1	5	1.0	1.0	1.1	8.9*	1.1	8.9*
		2	1	1.9	1.9				
	28	1	5	0.8	1.4	0.8	2.1	1.3	3.4
		2	1	0.8	0.8				
Constant Flow	2	1	5	0.5	0.9	0.5	4.3*	0.7	6.8*
		2	1	0.2	0.2				
	28	1	5	0.8	1.9	0.8	2.3	1.7	4.7
		2	1	1.0	1.0				

* Outside the acceptable limits

When the coefficient of variance of repeatability and reproducibility are compared with the repeatability and reproducibility limits defined in TS EN 196-1, it is observed that the repeatabilities and reproducibilities belonging to the constant water content and constant flow mortars are within the limits for 28 day compressive strength. Whereas, the repeatabilities and reproducibilities belonging to the constant water content and constant flow mortars are outside the limit for 2 day compressive strength.

According to repeatability and reproducibility values, the results of constant flow are better than those of constant water content for 2 day compressive strength.

5.4.5 Comparison According to Mann-Whitney Test

Again for CEM IV / B (P) 32.5R, the test results are compared by Mann-Whitney Test. The summary of the test results are given in Table 5.9. Although, it seems that there is not any difference in between TS EN 196-1 and constant flow test method for CEM IV / B (P) 32.5R, it must be noted that TS EN 196-1 test results belonging to laboratories 1 and 2 were discarded for constant water content mortar. Thus, when this situation is considered, the constant flow test method has given more similar test results than compared to TS EN 196-1 test method for pozzolanic cement.

Table 5.9 The Comparison of CEM IV / B (P) 32.5R Results

Mixes	Compressive Strength (Day)															
	2								28							
	Constant Water Content				Constant Flow				Constant Water Content				Constant Flow			
2	✓				✓				X				X			
3	X	✓			✓	✓			X	X			X	✓		
4	✓	X	✓		✓	✓	X		X	X	X		X	X	X	
5	✓	✓	✓	X	X	X	X	X	✓	X	X	✓	X	✓	✓	X
Laboratory Number	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4

✓denotes similar populations
X denotes different populations

CHAPTER 6

SUMMARY AND CONCLUSIONS

6.1 General

Two compressive strength test methods, following TS EN 196-1 (Constant Water Content) and ASTM (Constant Flow), were compared. For this purpose, two experimental studies were conducted. In the first part, compressive strength test of pozzolanic cement was performed in 16 different laboratories, which was organized by TCMA Council for Quality and Environment. In the second part, both TS EN 196-1 and constant flow test methods were conducted on portland, portland composite and pozzolanic cements in 5 different laboratories. The results obtained from mortar tests were examined by inter-laboratory test comparison and statistical analysis program SPSS. According to the findings, the following conclusions can be drawn:

- According to TCMA Council for Quality and Environment inter-laboratory test evaluation and statistical analysis program demonstrated that TS EN 196-1 is not an appropriate test method to determine the compressive strength of pozzolanic cement.
- Although there is not much difference in between 2 day compressive strengths obtained by TS EN 196-1 and constant flow test method, TS EN 196-1 test method is more appropriate for portland cements.
- According to results of CEM II / A-M (P-S) 42.5R type of cement obtained by both inter-laboratory test comparison and statistical analysis, there is not much difference in between TS EN 196-1 and constant flow for

portland composite cements. Thus, for portland composite cements, both TS EN 196-1 and constant flow tests are appropriate.

- According to results of CEM IV / B (P) 32.5R type of cement, for pozzolanic cements unlike for portland cements, constant flow test method is more appropriate than TS EN 196-1 test method, since the water to cement ratio is not appropriate to have a homogenous mix.

6.2 Recommendations for further studies

Considering the results obtained from this study, following recommendations for further studies are suggested:

- In previously performed inter-laboratory test comparisons mostly portland cements were used. Thus, it is recommended to perform these inter-laboratory tests by using different cement types.
- It is found that TS EN 196-1 test method is not appropriate for all cement types defined in TS EN 197-1. Thus, it is necessary to do some modifications on TS EN 196-1.
- In this study, only limited number of water to cement ratios were applied. Therefore, it is suggested to perform compressive strength tests on different types of cements having different water to cement ratio with more laboratories.
- In addition, in this study limited number of laboratories were performed the compressive strength test. Therefore, it is suggested to perform the tests with more laboratories.

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APPENDIX A

KOLMOGOROV-SMIRNOV TEST RESULTS

Table A.1 Kolmogorov-Smirnov Test Result of CEM IV / B (P-V) 32.5N

Number of Sample		13
Normal Parameters[a]	Mean	32.6
	Std. Deviation	5.3
Most Extreme Differences	Absolute	0.15
	Positive	0.15
	Negative	-0.15
Kolmogorov-Smirnov Z		0.55
Asymp. Sig. (2-tailed)		0.93

a. Test distribution is Normal.

Table A.2 Kolmogorov-Smirnov Test Results of CEM I 42.5R Having Constant Water Content

Compressive Strength (Days)		2	28
Number of Sample		6	6
Normal Parameters[a]	Mean	28.6	55.8
	Std. Deviation	2.02	2.59
Most Extreme Differences	Absolute	0.20	0.28
	Positive	0.20	0.26
	Negative	-0.13	-0.28
Kolmogorov-Smirnov Z		0.45	0.62
Asymp. Sig. (2-tailed)		0.99	0.83

a. Test distribution is Normal.

Table A.3 Kolmogorov-Smirnov Test Result of CEM I 42.5R Having Constant Flow

Compressive Strength (Days)		2	28
Number of Sample		5	6
Normal Parameters[a]	Mean	31.0	59.6
	Std. Deviation	2.10	2.89
Most Extreme Differences	Absolute	0.16	0.26
	Positive	0.13	0.24
	Negative	-0.16	-0.26
Kolmogorov-Smirnov Z		0.37	0.58
Asymp. Sig. (2-tailed)		0.99	0.88

a. Test distribution is Normal.

Table A.4 Kolmogorov-Smirnov Test Result of CEM II / A-M (P-S) 42.5R Having Constant Water Content

Compressive Strength (Days)		2	28
Number of Sample		6	6
Normal Parameters[a]	Mean	26.1	55.9
	Std. Deviation	1.62	2.13
Most Extreme Differences	Absolute	0.15	0.31
	Positive	0.15	0.26
	Negative	-0.15	-0.31
Kolmogorov-Smirnov Z		0.34	0.70
Asymp. Sig. (2-tailed)		1.00	0.71

a. Test distribution is Normal.

Table A.5 Kolmogorov-Smirnov Test Result of CEM II / A-M (P-S) 42.5R Having Constant Flow

Compressive Strength (Days)		2	28
Number of Sample		6	6
Normal Parameters[a]	Mean	26.7	57.1
	Std. Deviation	1.88	1.68
Most Extreme Differences	Absolute	0.21	0.27
	Positive	0.15	0.14
	Negative	-0.21	-0.27
Kolmogorov-Smirnov Z		0.47	0.60
Asymp. Sig. (2-tailed)		0.98	0.86

a. Test distribution is Normal.

Table A.6 Kolmogorov-Smirnov Test Result of CEM IV / B (P) 32.5R Having Constant Water Content

Compressive Strength (Days)		2	28
Number of Sample		3	6
Normal Parameters[a]	Mean	13.4	38.02
	Std. Deviation	0.70	1.44
Most Extreme Differences	Absolute	0.33	0.23
	Positive	0.33	0.19
	Negative	-0.24	-0.23
Kolmogorov-Smirnov Z		0.58	0.52
Asymp. Sig. (2-tailed)		0.89	0.95

a. Test distribution is Normal.

Table A.6 Kolmogorov-Smirnov Test Result of CEM IV / B (P) 32.5R Having Constant Flow

Compressive Strength (Days)		2	28
Number of Sample		6	6
Normal Parameters[a]	Mean	10.9	36.9
	Std. Deviation	0.68	2.06
Most Extreme Differences	Absolute	0.30	0.27
	Positive	0.25	0.27
	Negative	-0.30	-0.19
Kolmogorov-Smirnov Z		0.67	0.59
Asymp. Sig. (2-tailed)		0.76	0.87

a. Test distribution is Normal.

APPENDIX B

CHEMICAL AND PHYSICAL ANALYSIS OF CEMENTS

Table B.1 Chemical Composition and Physical Properties of the Cement Types

Compound (%)	CEM IV / B (P-V) 32.5N	CEM I 42,5R	CEM II / A-M (P-L) 42,5R	CEM IV / B (P) 32,5R
SiO2	33.60	19.45	23.46	26.66
Al2O3	8.85	5.41	6.63	7.63
Fe2O3	4.93	3.48	3.36	3.43
CaO	40.84	64.13	58.37	51.,18
MgO	1.96	1.50	1.63	1.81
SO3	2.99	3.05	2.60	1.82
K2O	1.1	0.57	0.67	0.83
Na2O	0.65	0.16	0.13	0.02
Free CaO	0.56	0.74	0.56	0.24
LOI	5.05	1.69	2.72	6.21
Cum. % Retained on 45µ	-	4.7	3.8	3.6
Specific Gravity	-	3.14	3.04	2.88
Blaine Fineness (cm2/g)	-	4230	4390	4680

Table B.2 Main Constituents of the Cement Types

	Main Constituents (%)				
Cement Type	**Klinker**	**Tras**	**Fly Ash**	**Limestone**	**Slag**
CEM IV / B (P-V) 32.5N	56.0	18.0	22.0	4.0	-
CEM I 42.5R	95.0	-	-	5.0	-
CEM II / A-M (P-S) 42.5R	86.0	7.0	-	3.0	4.0
CEM IV / B (P) 32.5R	60.0	38.0	-	2.0	-

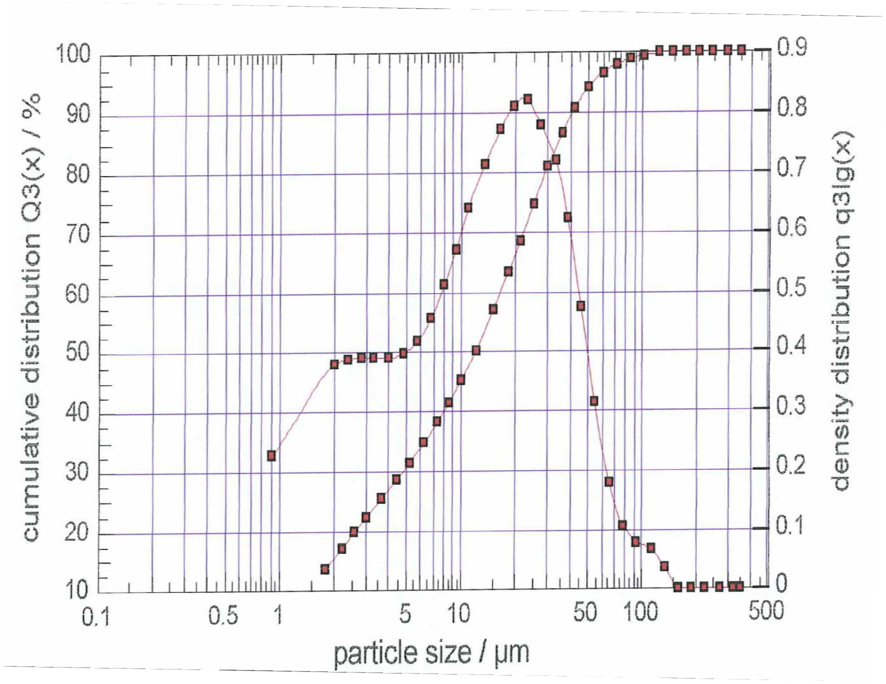

Figure B.1 Particle size distribution of CEM I 42.5R

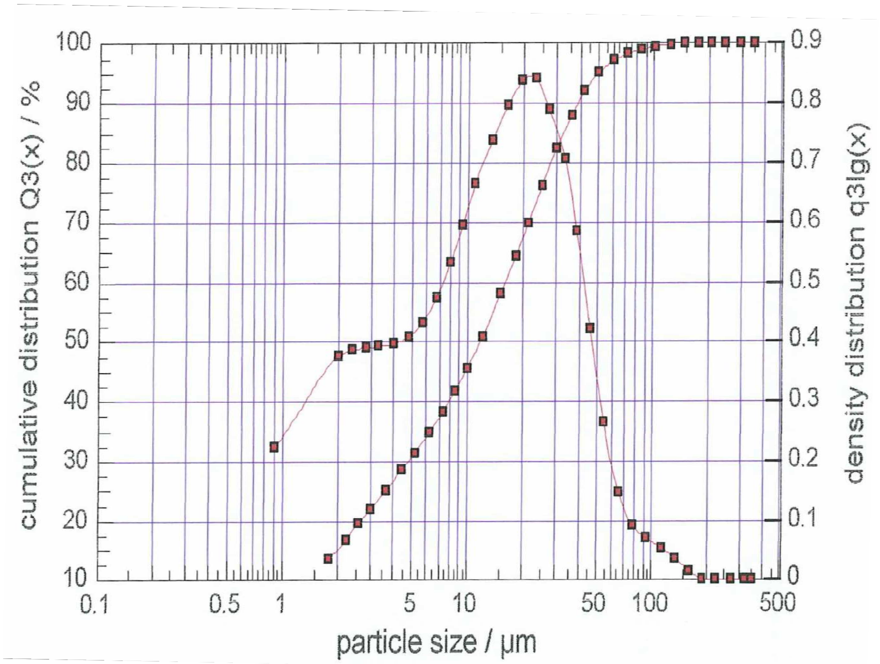

Figure B.2 Particle size distribution of CEM II / A-M (P-S) 42.5R

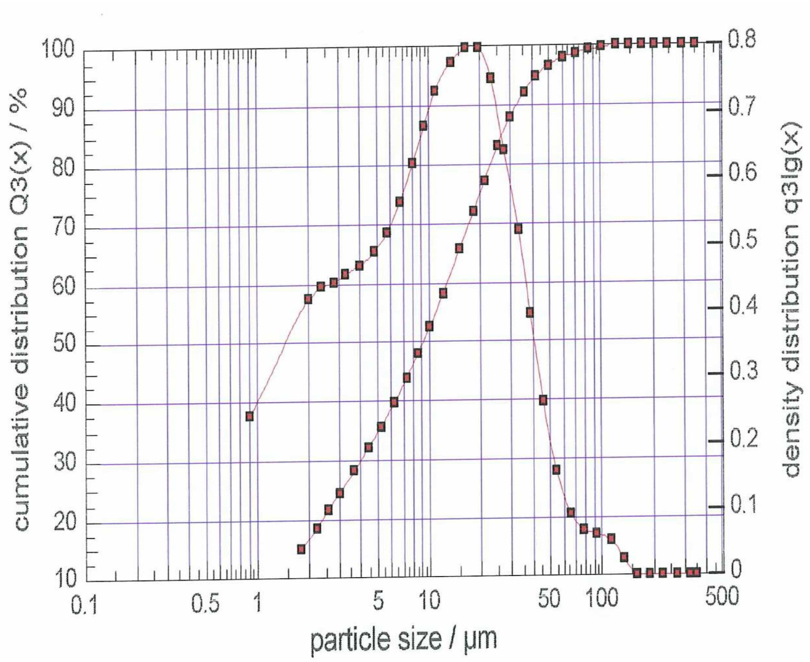

Figure B.3 Particle size distribution of CEM IV / B (P) 32.5R